LABORATORY APPARATUS AND MATERIALS EXHIBITION
PICTORIAL SUPPLEMENT

LABORATORY PRACTICE

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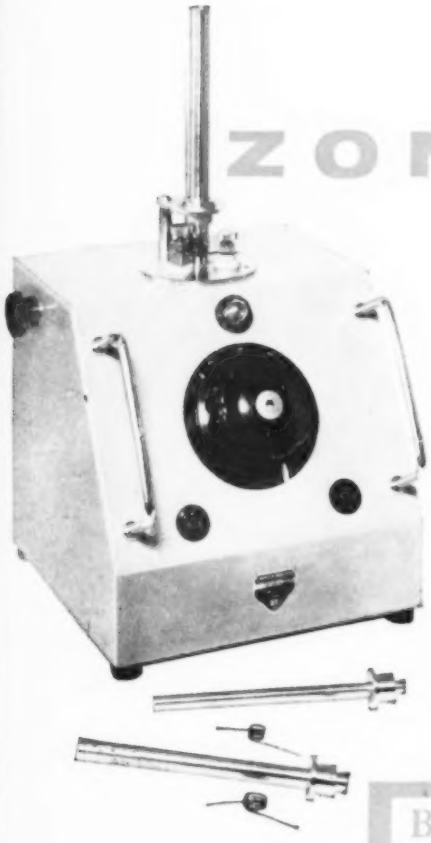
VOL. 9 No. 8 Published Monthly AUGUST 1960



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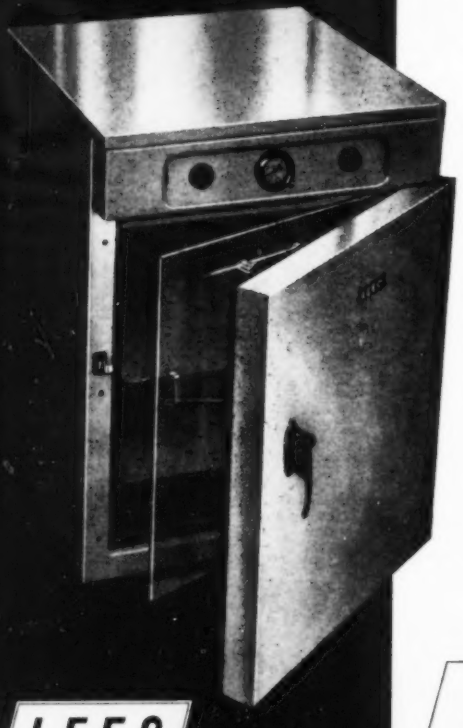

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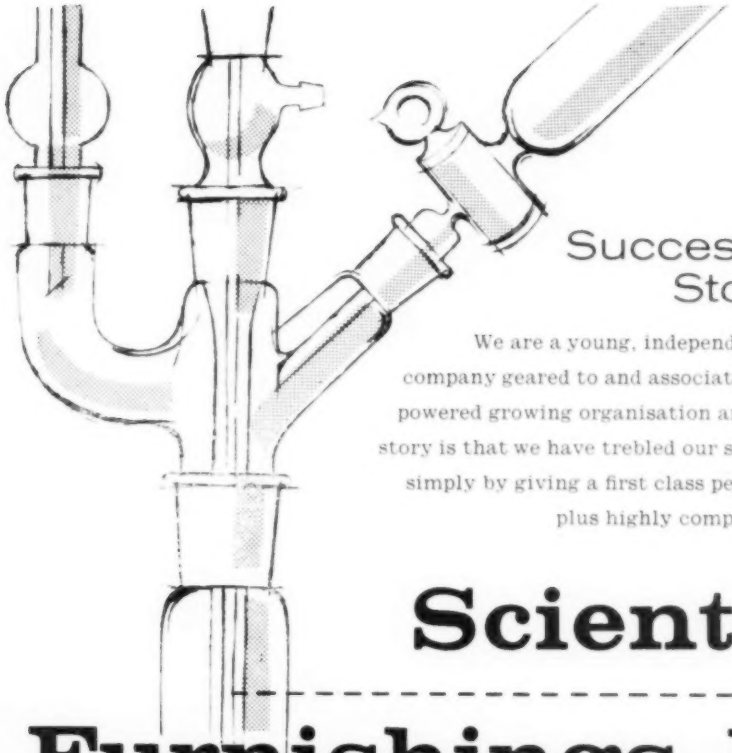

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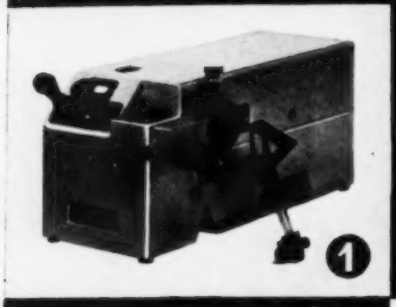

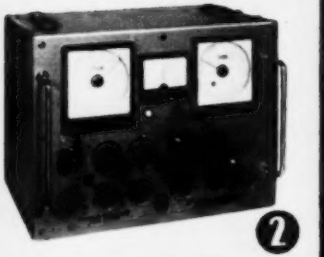

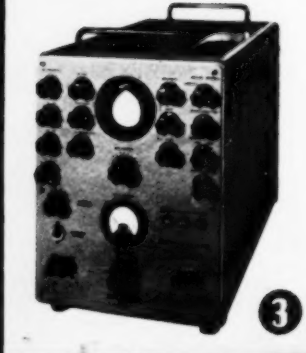

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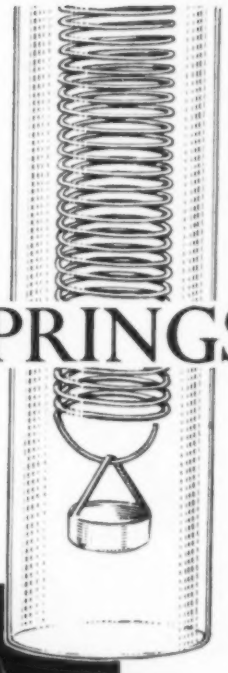

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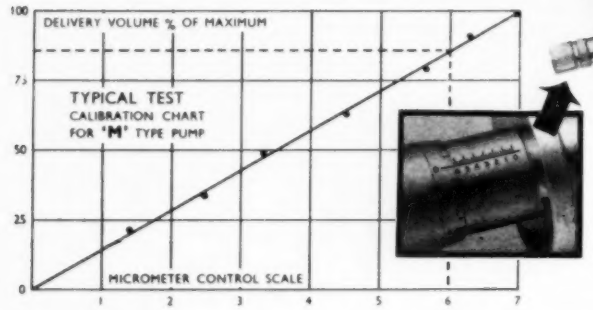

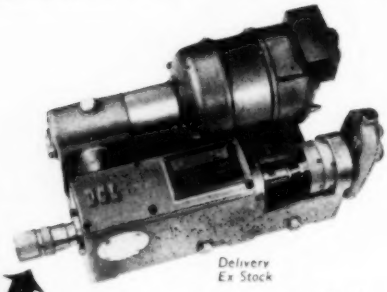

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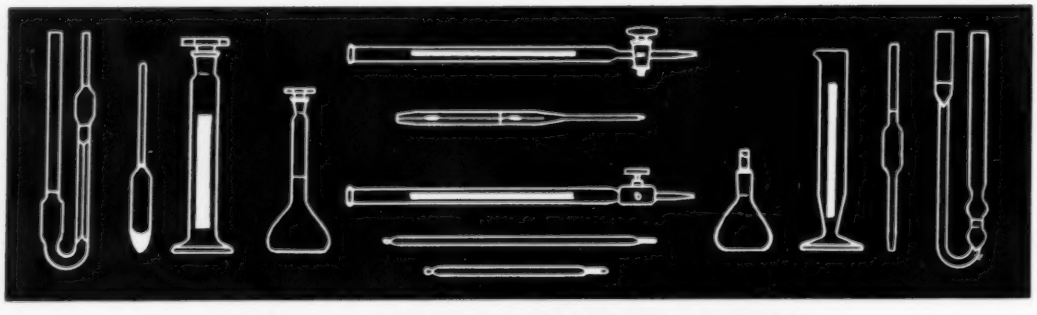

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In fact, it is the Analytical Chemist's ceaseless search for improved techniques that makes these higher standards realistic, as no legislation or published standard can itself ensure purity unless an analytical method exists which is sufficiently sensitive and accurate on all occasions, and for which there is the instrument aid available to make it practically useful. For companies engaged in the development of instruments aimed at making the Analytical Chemist's task lighter, it is very difficult to talk to enough people in the field to find out what is nearest and dearest to their hearts in this respect. However, Southern Instruments Limited of Camberley have set themselves this task and have recruited a team to tackle it. Initially concentrating in the electro-chemical field, this company is building up a range of aids which will be designed to provide the benefits of convenient operation, saving in time and money, and the employment of staff appropriate to the job in question. Their fully equipped laboratory led by J. Hetman, F.R.I.C., is constantly and confidentially dealing with a vast range of Analytical matters for Analysts who feel, for example, that Polarography may have advantages to offer them. This enables the chemist to be fully satisfied that his requirements are met beforehand. This service is free, and open to anyone, and whilst it is not claimed that Southern Instruments know all the answers, they do know many of them. Future electrochemical products will also be backed by an Applications Advisory Service.

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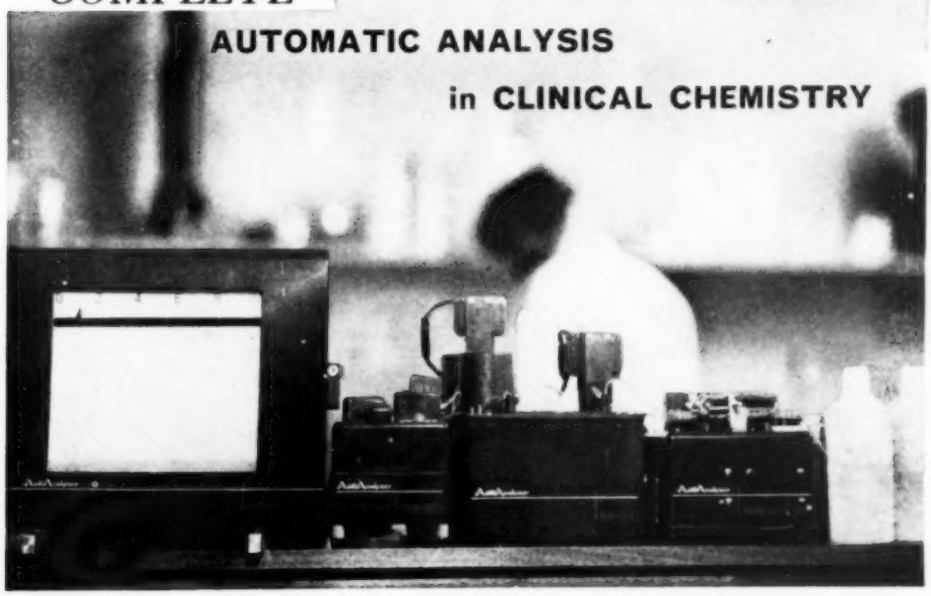

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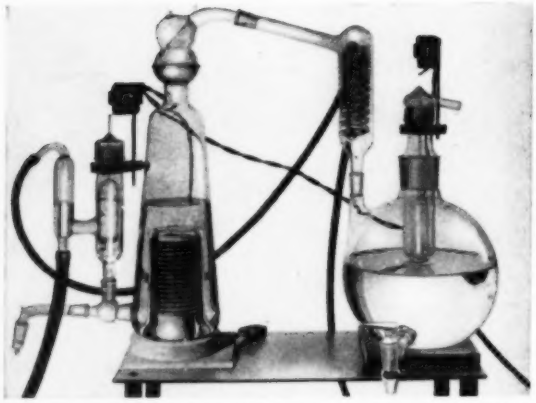

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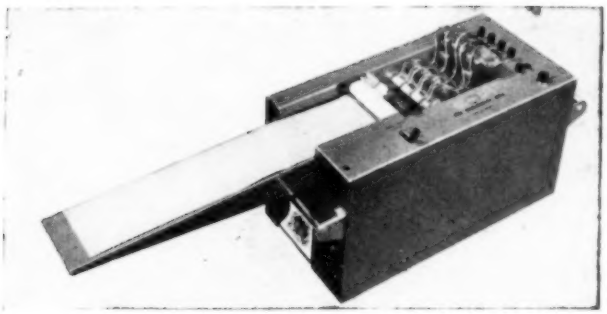

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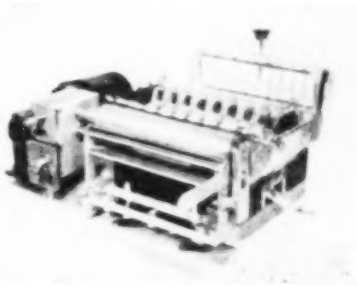

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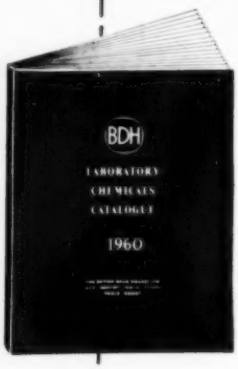

this new edition . . .

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Section 3 of the catalogue, formerly limited to amino acids, now also contain reference lists of other biochemical materials grouped for convenience in their various classes of carbohydrates, lipids, proteins, sterols, etc.

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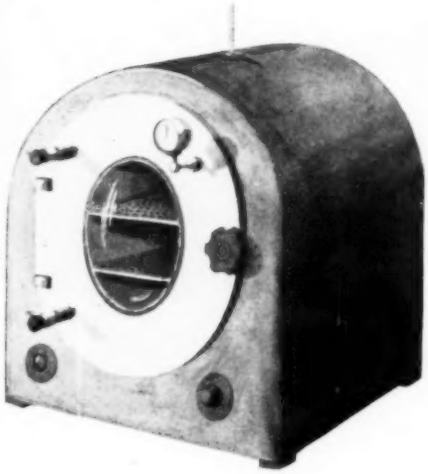

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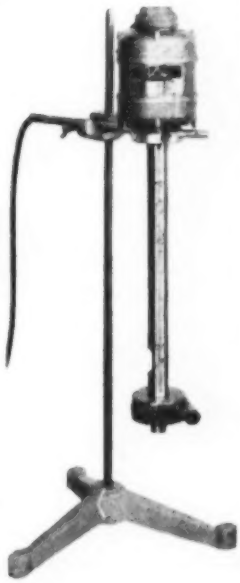

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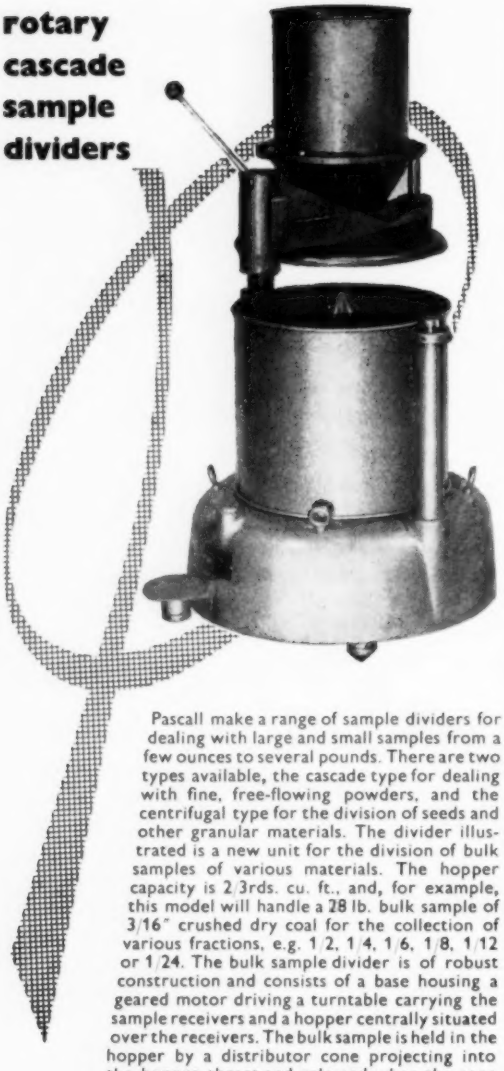

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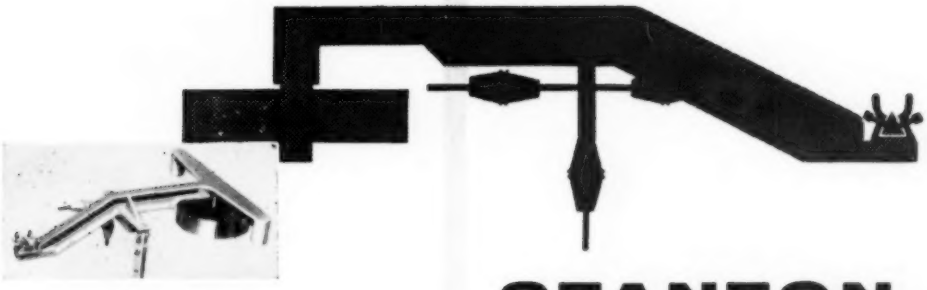

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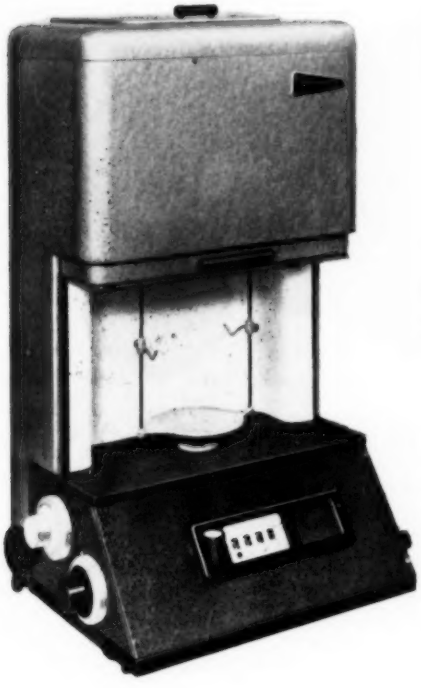

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EDITORIAL

Pictures from an Exhibition

SOME years ago the Russian composer Moussorgsky visited an exhibition and afterwards wrote a pianoforte suite to convey his impressions of ten pictures by the architect Hartmann. As a visitor to the recent exhibition of apparatus and materials we, too, may record our impressions of this first effort, supplemented by discussions with other visitors.

The feature which seems to have impressed itself most strongly was the very friendly and informal atmosphere about the whole venture. Scientists were able to use it rather like the *salon* of the 19th century—to meet their friends, discuss problems of mutual concern, to go somewhere with a definite objective which had an intellectual interest, and in general to spend their time usefully and in an enjoyable manner. Coupled with this aspect was the unanimous verdict that it supplied a long-felt want, a hackneyed phrase but one nevertheless which could be said truthfully. There have been well-organized exhibitions of specialist interests, and small scale exhibitions organized by individual firms, but never before has there been an exhibition on such general lines, where one could wander round at leisure and see virtually every type of scientific apparatus.

The firms displaying their equipment had the good sense to see that only technically knowledgeable staff were on the stands (actually many were qualified scientists) and this was a facet much appreciated by the visitors. There is nothing more annoying than to go to an exhibition and find that the immaculately attired young man or excessively 'got up' young woman is quite unable to answer a straight-forward question about the goods being exhibited. In modern business it is not enough to be willing; one must be properly equipped. The exhibiting firms in turn also seemed to be abundantly satisfied, and presumably their opinion is based on tangible experimental results beyond the criticism of the most fastidious statistician.

While on this question of salesmanship may we comment on the growing pernicious system of advertising scientific equipment by circulars and in the journals without mentioning price? We ourselves are a member of a number of scientific societies, and we

receive replicate circulars from one firm making the most enthusiastic claims for their goods but giving no mention of price. These circulars go straight into the waste box, so makers of scientific equipment—please note! The scientist who is thinking of buying apparatus asks two questions—Will it do what I want it to do? How much does it cost?

Nothing is ever complete or perfect in this world and it would be surprising if absolutely no criticism could be made of this exhibition. However, the only valid one seems to be that a few firms, and consequently a few specialized types of equipment, were absent. There is a very simple explanation for this. Most firms plan their exhibition commitments a long time in advance. This exhibition was organized at relatively short notice, and unfortunately a few firms were unable to include this exhibition in their programme. There is little doubt that this difficulty will not occur again.

The special lectures were a novel and warmly welcomed part of the exhibition. We understand that most of them could have been 'sold out' two or three times, and that as many as 900 applications were received for tickets for one of them. The lecturers were of the highest calibre, men of considerable reputation in their own fields, and the demonstrations in particular were excellent.

Summing up, this exhibition enabled the ordinary working scientist to get a bird's-eye view of modern scientific apparatus and materials, and to discuss them with experts, in the space of a few hours. Without such an exhibition it would take many days and several journeys and much more expense to achieve the same object.

We hope that next year the exhibition will be held again, with increased coverage, particularly of highly specialized apparatus and the latest books. It is worth remembering that books, and especially laboratory manuals, are as much tools of our trade as apparatus and chemicals.

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THE APPLICATION OF THE CATHODE RAY POLAROGRAPH IN FOOD ANALYSIS

by J. S. Hetman

Southern Analytical Ltd., Camberley, Surrey

THE sale of food in Great Britain is controlled by regulations recommended by the food standards committee. The general limits for copper, lead and zinc laid out in Bell's Sale of Food & Drugs Act (1956) are given in Table I.

The present work describes polarographical methods for the simultaneous determination of Cu, Pb and Zn in various foods and drinks. The estimation of vitamin C, saccharin, and sulphur dioxide in a variety of soft drinks and fruit juices is also described.

For the accurate estimation of any of these elements by classical methods, very tedious analytical operations are required and the time involved to complete the analysis is about three or four hours for Cu and two to three days for all three metals. In general the samples are decomposed by ashing or by wet decomposition. The metals under consideration are extracted by organic solvents, or precipitation by H_2S, and determined colorimetrically.

TABLE I

Recommended limits for Cu, Pb and Zn

Element	*Limits p.p.m.*	
	In Beverages	*In Other Foods*
Cu	2	20
Pb	0·2	2
Zn	5	50

Mr. Hetman delivers his lecture (chairman: Dr. J. D. Mounfield).

A number of papers have been published dealing with the polarographical determination of Cu, Pb and Zn[1–4]. Prior to polarographical analysis, dry ashing or wet decomposition is necessary, which in no way differs from the procedure for classical methods. Using conventional polarographs, it is essential to apply chemical separation to concentrate the metals being determined, and to remove the interference of one upon another (Cu must be removed for the determination of Pb, Fe interferes in the determinations of Cu and Zn).

In this work a new technique has been developed which enables the simultaneous determination of Cu, Pb and Zn in one to two hours. A cathode ray polarograph is used for the estimation of each metal. The high sensitivity and resolution power of the instrument eliminates the necessity of preliminary chemical separation.

Work has also been carried out in the estimation of vitamin C, saccharin, and sulphur dioxide in a variety of soft drinks and fruit juices. In accordance with the Food Labelling Act, the minimum vitamin C content must be expressed on the label. In a

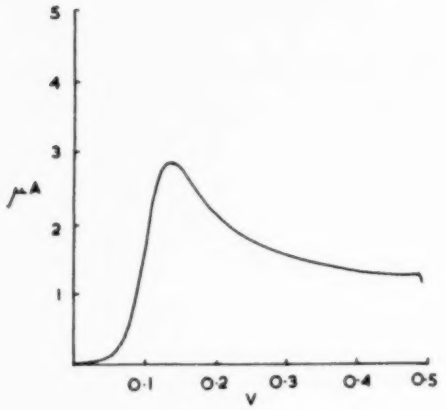

FIG. 1. TYPICAL Cu WAVE 3 μg/ml

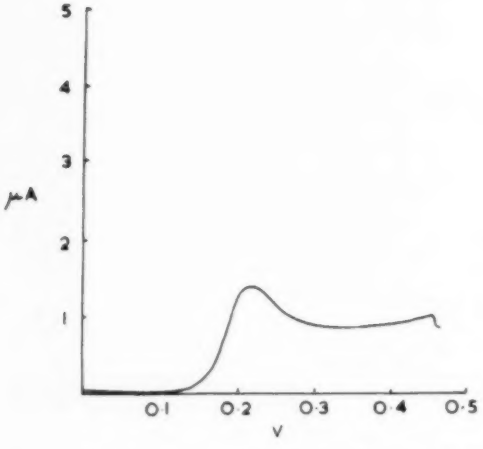

FIG. 2. TYPICAL Pb WAVE 0·4 μg/ml

recent Food Standards Committee Report on soft drinks, the use of saccharin is prohibited except for diabetic drinks. Food preservatives such as sulphites and benzoates are used in wide ranges of concentration and methods for their rapid estimation have been investigated.

Experimental

In a base electrolyte composed of 0·2M tartaric acid in 0·1M HNO_3, Cu, Pb and Zn can be determined simultaneously. The presence of stannic tin has no effect on the waves of Cu, Pb or Zn. Well shaped reproducible waves are formed with the peak potentials for Cu at —0·25V and Pb at —0·6V applying cathodic reduction and Zn at —1·2V applying anodic oxidation.

Known amounts of Cu, Pb and Zn were introduced into 10 ml. volumetric flasks, made up to volume with base electrolyte, shaken well and the polarograms recorded on 5 ml. aliquots, after de-aerating with N2 for 3 min. Derivative display was applied to obtain the Zn wave. (Fig. 1-3). The wave heights obtained are summarized in Table II.

TABLE II

Wave heights of Cu, Pb and Zn

Metal	*Scale Factor*	*Metal added μg/ml.*	*Wave Height Divisions of Graticule*
Cu	0·6	1	9
		2	17
		5	43
Pb	0·15	0·2	7
		0·4	14
		0·6	21
Zn	0·06	5	14
		10	28
		15	41

Analysis of Cocoa

Two g. of sample in a silica crucible, capacity 25 ml., were charred and ignited at a temperature not exceeding 500 C. The ash was dissolved

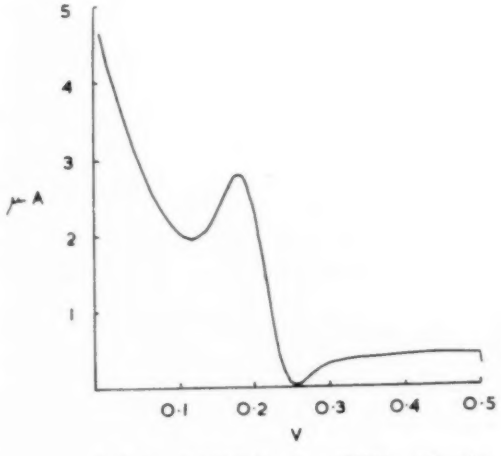

FIG. 3. TYPICAL Zn WAVE 10 μg/ml

in 1 ml. of 1:1 HNO_3, the solution evaporated to dryness, the residue dissolved in base electrolyte, boiled, transferred into a 10 ml. flask, made up to the mark and filtered through a dry folded filter paper. The polarograms were recorded on a 5 ml. of filtrate and the concentrations of each metal obtained from a calibration graph prepared as stated above. Cu 9 p.p.m., Pb 0·5 p.p.m., Zn 60 p.p.m. The results obtained are in agreement with classical methods. (Table III).

Determination of Copper and Zinc in Beer

Ten ml. of sample were placed in a silica crucible, capacity 25 ml., evaporated to dryness, charred and ignited at 600-700 C. The residue was dissolved in 1 ml. 1:1 HNO_3 and the solution evaporated to dryness. Five ml. of tartaric acid nitric acid base electrolyte were added to the dry residue, the solution boiled, transferred into a 10 ml. flask, made up to volume with base electrolyte. The polarograms were recorded on a 5 ml. aliquot after de-aerating with N_2, on start potential —0·1V for Cu, applying cathodic reduction, and —1·35V for Zn, applying anodic oxidation and derivative display. The concentrations were obtained from a calibration graph, prepared from synthetic solutions.

The above method is applicable for samples in which the Fe content is not greatly in excess of the Cu. If the Fe is more than 50 times the concentration of Cu, interference is observed in the Cu wave and a HCl/ascorbic acid base electrolyte is preferable. In this base electrolyte Fe is reduced by the ascorbic acid and concentrations of Fe up to 0·5 per cent show no interference.

Simultaneous Determination of Copper, Lead and Zinc in Meat Extract

Two g. of sample were weighed in a silica crucible, charred and ignited at a temperature not exceeding 500 C. The ash was dissolved in 2 ml. of conc. HCl, oxidized with 0·5 ml. of conc. HNO_3 and evaporated to dryness. Two ml. of conc. HCl were added to the dry residue and the evaporation repeated. Finally 2 ml. of conc. HCl were added, followed by 5 ml. of water, the solution boiled for 2-3 min. cooled and transferred into a 20 ml. flask. 0·2 g. of ascorbic acid was added to the flask, the solution made up to the mark with water and shaken well. The polarograms were recorded on a 5 ml. aliquot, on start potential —0·07V for Cu and Pb, applying cathodic reduction and —1·3V for Zn, applying anodic oxidation and derivative display. The concentrations, Cu 4 p.p.m., Pb 4 p.p.m. and Zn 40 p.p.m. were obtained from a calibration graph. The concentrations found by classical methods were in good agreement. No Cu or Pb was found in the reagent blank, Zn was detected but was negligible (0·1 μg/ml.).

Simultaneous Determination of Copper, Lead and Zinc in Wine

Ten ml. of sample were evaporated to dryness, charred and ignited at a temperature not exceeding 500 C. The residual ash was dissolved in 1 ml. of conc. HCl, digested for 2-3 min., approximately 2 ml. of water added, the solution boiled, cooled and transferred into a 10 ml. flask. 0·1 g. of ascorbic acid was added, the solution made up to the mark with water, filtered through a dry folded filter paper, Whatman No. 40, and the polaro-

TABLE III

Concentrations of Copper, Lead and Zinc in Various Foods and Drink

Sample	Cu		Pb		Zn	
	Pol. Method*	*Class.* Method*	*Pol. Method*	*Class. Method*	*Pol. Method*	*Class. Method*
Cocoa	9	11	0·5	0·4	60	50
Beer	8	9	—	—	1·7	2·0
Meat Extract	4	4	4	5	40	45
Wine	0·15	0·2	0·1	0·05	3	3

**Pol.* — *Polarographical* **Class.* — *Classical.*

grams recorded on 5 ml. of filtrate. Cu was recorded at start potential —0·1V and Pb at —0·3V using cathodic reduction; Zn was found using start potential —1·25V by anodic oxidation with derivative display.

In the HCl/ascorbic acid base electrolyte the presence of Sn has no effect, if its concentration is not more than twice that of Pb. If Sn is in greater excess the removal of Sn with a HBr/Br mixture is essential. The operation is relatively simple.

To the residue, after ashing the sample, is added 0·5 ml. of HBr saturated with Br, and the solution evaporated to dryness on a water bath. One ml. of conc. HCl is added, followed by 5 ml. of water, the solution boiled for 2-3 min., cooled and transferred into a 10 ml. flask. 0·1 g. of ascorbic acid is added and the solution made up to the mark with water. The polarograms of Cu, Pb and Zn are obtained as previously described.

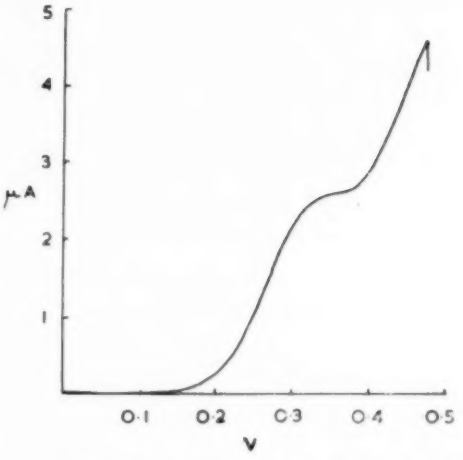

FIG.5. SACCHARIN IN LEMON CORDIAL

Determination of Vitamin C in Soft Drinks

In a phosphate buffer, pH 8, vitamin C is oxidizable with the formation of an anodic wave with peak potential at + 0·1V. The linear relation between wave height and concentration is always preserved and no interference from the other components of soft drinks manufactured in this country has been observed.

In soft drinks, ascorbic acid is added during manufacture, therefore no pretreatment of the sample to convert combined ascorbic acid to free ascorbic acid is necessary.

The determination is one of the easiest and up to 100 samples per day could be analysed.

The buffer solution is prepared by adding drop by drop a 10 per cent solution of KH_2PO_4 to a 2 per cent solution of Na_2HPO_4, 12 H_2O until the pH reached eight units. According to the amount of ascorbic acid present, 0·5 to 2 ml. of sample are introduced into a 10 ml. flask, made up to volume with the buffer solution and the polarogram recorded on 5 ml. on start potential —0·2V The concentration is obtained from a calibration graph prepared from AnalaR grade ascorbic acid. Fig. 4 is a typical polarogram of vitamin C in a lemon cordial (1·6 μg/ml.). Scale factor 4·0.

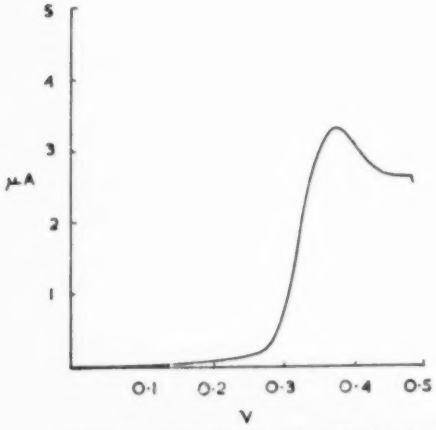

FIG.4. VITAMIN C IN LEMON CORDIAL

Determination of Saccharin in Soft Drinks

Saccharin is reducible with the formation of a single wave at peak potential —1·25V. In some samples, the concentration can be determined directly on the solution, placing the sample in the cell, de-aerating, and recording the polarogram. In others, an HCl/LiCl electrolyte is used (250 ml. of 0·2N HCl, 500 ml. of 0·1 N LiCl, pH 1·0).

2·5 ml. of sample are introduced into a 10 ml. volumetric flask, brought up to the mark with base electrolyte and the polarogram recorded on a 5 ml. aliquot, on start potential —0·9V. In many samples the saccharin wave is reduced very close to that of hydrogen or of other components present in the soft drink and measurement of the saccharin wave on direct current is impossible, therefore derivative current is used. A linear relation between wave height and concentration exists over a wide range of concentration. The saccharin contents are obtained from a calibration graph.

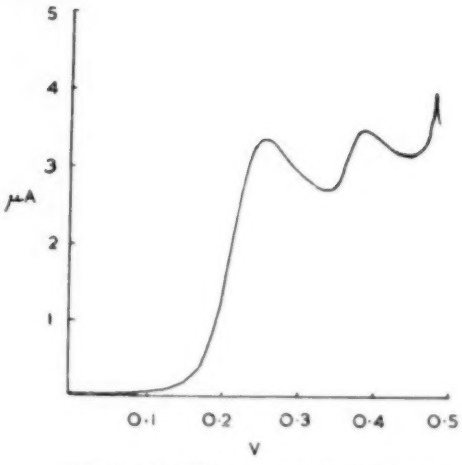

FIG. 6. SULPHITE IN LEMON CORDIAL

Fig. 5 is the polarogram of saccharin in a lemon cordial 2 μg/ml. Scale factor 15·0.

Determination of Sulphur Dioxide in Drinks

Sulphites are oxidizable in an acid medium with the formation of a double wave at peak potentials —0·25 and —0·15V.

Acidifying the sample will introduce the possibility of losing SO_2 on de-aeration, therefore the sample is first made alkaline with NaOH, de-aerated, acidified with oxygen-free HCl, and the polarogram recorded. Linearity between wave height and concentration is always preserved under constant acidity conditions.

For the determination of sulphites in lemon cordial, 2 ml. of sample are introduced into a 10 ml. flask, and made up to the mark with a base electrolyte consisting of 1 per cent KCl in 0·1N NaOH. Five ml. of solution are transferred into a polarographic cell, and de-aerated with N_2. 0·2 ml. of conc. HCl are added, the solution mixed by passing five or six bubbles of N_2 and the polarogram recorded on start potential —0·55V, applying anodic oxidation (Fig. 6). The concentration of sulphites is obtained from a calibration graph.

Determination of Benzoic Acid in Soft Drinks

Benzoic acid is reducible in a base electrolyte of 0·3M tetraethylammonium bromide with the formation of a single wave at peak potential —1·7V. A direct determination of benzoic acid, by mixing the sample with base electrolyte is not possible, due to interference from other substances reducible on or near the same potential. A variation of the pH in various ranges gives no improvement.

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[4]Polarography in Germany, B.I.O.S. Final Report No. 1458 p. 24.

Water Pollution Research

Only six years after it was opened, the Water Pollution Research Laboratory at Stevenage is already being extended. Work has started on a new wing, scheduled for completion early next year, which will accommodate an additional 40 research staff and increase the total research effort of the Laboratory by nearly 50 per cent. This is announced in the Laboratory's annual report, published for D.S.I.R., by H.M.S.O. (price 7s. 6d.) Such rapid expansion has been called for by the swiftly increasing use of water in Britain, especially for industrial purposes, making it more and more necessary to use waters to which industrial and sewage effluents have already been added. Water can only be re-used satisfactorily if polluting liquors are adequately purified before discharge, and the research undertaken by the Laboratory is therefore of growing importance in the national economy.

Most of the new accommodation at Stevenage will consist of pilot-scale laboratories in which new experimental methods of treating waste liquors can be taken to the stage where they can easily be introduced into full-scale practice.

A large part of the Laboratory's present work is concerned with basic problems in the treatment of sewage and industrial waste waters. A new experiment was started last year in a large pilot plant in the open air, in which four of the types of medium now most commonly used in percolating filters (clinker, blast furnace slag, broken rock, and gravel), each in two sizes, are being used to treat purely domestic sewage under controlled conditions. Using radioactive tracers, efficiency is being correlated with period of retention in these filters.

Work has continued during the year to determine the relative efficiency of aeration in the activated-sludge process by different means, including diffused air, rotating brushes, and Simplex aeration units, and with the effect of various factors, including the presence of synthetic detergents, on performance.

With the aim of solving the numerous public health problems caused by the introduction of synthetic detergents, large-scale tests on a material have been carried out during the year for the Standing Technical Committee on Synthetic Detergents. First results show that household washing powders, containing the new, 'soft' form of surface-active material, developed by the manufacturers, are more easily decomposed by bacterial action.

Further work has been done on the toxicity to fish of a number of substances, including ammonia, gas liquors, and salts of lead, copper, and zinc. First results of a series of experiments on the effects of inert suspended solids on fish show that over a period of some three months, concentrations of about 100 p.p.m. may kill trout. The suspended material appears to bring about pathological changes in the gills.

Among the instruments being developed are an automatic recorder for suspended solids in sewage effluents, an automatic sampler, and an apparatus for determining the concentration of dissolved oxygen in laboratory samples; this replaces the usual chemical procedure for oxygen determinations.

Prof. R. Belcher.

INDICATORS

by R. Belcher

Professor of Analytical Chemistry, Birmingham University

DESPITE the great advances in instrumentation and instrumental methods for end-point detection, visual indicators remain the most widely used reagents in analytical chemistry and the search for new indicators continues on an unprecedented scale.

Indicators are used in acid-base, precipitation, redox and complexometric titrations and examples of each class are considered under these headings.

Acid-base Indicators

The conventional acid-base indicators which are available are generally satisfactory and there is little need for improvement. However, some indicators have been described which may be of service in special cases. For example, Verma and Agarwal[1] have described a screened-mixed indicator which can be used in the titration of the first two stages of phosphoric acid. It consists of methyl orange, Xylene Cyanol FF and phenolphthalein; the colour changes occur at pH 4·5 and pH 9·0. It is usually necessary to titrate separate aliquots but with this composite indicator both titrations can be done on the same solution.

An interesting series of mixed indicators has been described by Mikhailov[2]. These give a colourless zone at the end-point; the ranges on either side of this colourless zone are coloured. An example of these mixed indicators is hexamethyoxytriphenylcarbinol-thymolphthalein (equal parts of 0·1 per cent ethanolic solutions). The colour is red between pH 0 and 4·6, colourless to 9·3 and blue above 9·3.

For titration in coloured solutions, nitron hydrochloride has been proposed[3]; chloroform is added and the colour change takes place in the organic phase.

In the early days of titrimetric analysis inorganic indicators were quite common, but were rapidly superseded by organic indicators. More recently, some inorganic indicators have been described for special purposes and have been referred to in several monographs[4]. These indicators sometimes have advantages over organic indicators when a titration has to be done in an oxidizing medium which would destroy an organic indicator. The most recent example of this type of indicator is yellow basic ferric sulphate[5], which is converted into red iso-orthoferrihydroxide by hydroxyl ion at about pH 8. The indicator is prepared by oxidizing ferrous sulphate with hydrogen peroxide.

Perhaps the most interesting of the recent acid-base indicators are the chemiluminescent indicators. These are indicators which emit light at the end-point. The first indicator of this type was described by Kenny and Kurtz[6]. More recent studies have been carried out by Erdey and his co-workers in Budapest. Examples of this class of indicator are lophine, luminol and lucigenin. There are several papers available describing the use of these indicators[7]; it is also possible to apply them in argentometric, redox and complexometric titrations[8].

Precipitation Indicators

Indicators of this type are generally used in argentometry. Many new adsorption indicators have been proposed but they have not come into wide use because of their limitations when electrolytes other than halides are present. Mehlig[9] has recommended potassium rhodizonate as an argentometric indicator. This compound was recommended many years ago as an indicator for the titration of sulphate with barium salts, but is now little used because of the poor end-point. It would seem, however, that the reaction with silver ions is much more sensitive, for Mehlig claims that the indicator has advantages over chromate and thio-

This lecture was given under the chairmanship of Prof. F. C. Tompkins.

cynate when used in the Mohr and Volhard titrations respectively.

Cyanide is usually titrated with silver nitrate according to the Liebig-Denigés procedure; a turbidimetric end-point is used. Several indicators depending on colour-change have been suggested but their use does not appear to be widespread. Recently Bhatki[10] has recommended the titration of cyanide with nickel salts; he uses as indicator resacetophenone-oxime which gives a yellow-green turbidity at the end-point.

Redox Indicators

This is a field which has expanded considerably since Knop first recommended diphenylamine as a redox indicator for use in titrations of iron(II) with dichromate. The abnormal behaviour of some of those indicators is still unexplained. Because diphenylamine is first oxidized to diphenylbenzidine, the latter compound has been used in place of diphenylamine in order to reduce the blank value. Unfortunately, when diphenylbenzidine is added to a test solution it is precipitated—owing to its insolubility in aqueous media—and is then difficult to oxidize; over-titration may result. When diphenylbenzidine is formed *in situ* by oxidation of diphenylamine this effect is not noticed. An improvement on both these indicators is diphenylamine-sulfonic acid[11] which does not yield precipitates with ions such as tungstate, molybdate, etc. This indicator is perhaps the one most widely used for titrations with dichromate.

It is surprising that the work was never carried to its logical conclusion. It would be expected that diphenylbenzidine-sulfonic acid would be the best of this group, because it should have a lower blank value than the corresponding diphenylamine-sulfonic acid and should not precipitate like the parent base. This indicator was in fact described in 1935[12] but the work appears to have passed unnoticed. Recently, the material has become commercially available. It has a lower blank value than diphenylamine-sulfonic acid, as would be expected, and the end-points are very sharp. In our opinion[13], this indicator is the best of the diphenylamine group.

A few years after the development of redox indicators, the independent studies of Willard and of Furman in the U.S.A. placed ceric sulphate on a sound basis as a oxidimetric titrant. In the early days of this titrant there were no satisfactory indicators available and it was not until Kunz[14] had established the redox potential of the system that it became apparent that an indicator of higher redox potential than the diphenylamine type was required. The work of Walden, Hammett and Chapman[15] resulted in ferroin being recommended. The introduction of ferroin was a great advance and this indicator has been called the 'phenolphthalein of oxidimetry'[16]. Ferroin has a redox potential of 1·06 volts and it is surprising to find even today some curious statements about its applications in the text-books. This is possibly due to incomplete understanding of the early literature on the subject. Sometimes ferroin is advocated as indicator for titrations of iron(II) with dichromate, yet under the normal conditions of this titration it is not possible to obtain an end-point. It is an easy matter to see the reason if the redox potentials of the system and indicator are considered. The reaction between dichromate and iron(II) follows an unusual course. When dichromate is titrated into iron(II), the equivalence potential ranges from 0·85-1·0 volts; this is below the redox potential of ferroin and therefore no end-point is obtained. In the reverse titration, however, a curious phenomenon occurs and the equivalence potential ranges from 1·22-0·85 v. In the reverse titration, therefore, ferroin functions satisfactorily. It is also satisfactory in the direct titration with dichromate when the acidity of the solution is raised to about 4 M in sulphuric acid. The redox potential of the system is increased and that of the indicator is decreased at this acidity.

We were puzzled for many years by the indicator *N*-phenylanthranilic acid[17]. The redox potential is reported as being 1·08 v. and it is usual to find the statement made that it will serve both in dichromate and ceric sulphate titrations. Because this potential is even higher than that of ferroin, it is surprising to find that it does in fact give an end-point in both titrations. This led us to redetermine the transition potential of this indicator some little time ago[18]. We have found the potential is 0·89 v. which is the potential to be expected for an indicator which functions with both titrants. It would be very surprising if the potential of *N*-phenylanthranilic acid were as high as originally reported. The actual structure is diphenylamine-*o*-carboxylic acid; the potential of diphenylamine is 0·76 v. and it is hardly to be expected that the introduction of a carboxyl group would raise the potential to such a degree.

The greatest contributions which have been made in the field of redox indicators are those of Prof. G. F. Smith and his co-workers. He has synthesized many substituted ferroins and dipyridyls and has determined their redox potentials. As a result of these studies ferroin type indicators are available covering the range 0·86-1·25 v. Smith and Brandt[19] have recommended 5:6-dimethylferroin as indicator in titrations with dichromate; the redox potential is 0·97 v. This indicator undoubtedly works well in this system but in our opinion is inferior to diphenylbenzidine sulfonic acid. It is known that the redox potential of 2-2-dipyridyl is also 0·97 v. and it would therefore be expected to behave like 5:6-dimethylferroin with dichromate, but no end-point is obtained. It would seem then that the suitability of a redox indicator for a particular system cannot be assessed completely by its redox potential.

The two indicators which have the highest potential of the series are nitroferroin and ruthenium 2-2'-dipyridyl. Their redox potentials are both 1·25 v. and they are suitable for use in titrations with ceric sulphate in perchloric acid medium. This reagent has the highest redox potential of any known titrant and ferroin itself

is unsuitable because its potential is too low. Another puzzling feature which still awaits explanation has come to light from recent work of Rao[20]. Rao recommends in place of the two preceding indicators for ceric-perchloric acid titrations, certain triphenylmethane dyestuffs such as erioglaucin A and eriogreen B. These indicators were used in the early days of ceric sulphate titrations before ferroin was discovered and their redox potentials are well known (1·0 v.). This conflicts with the current belief that ferroin is unsuitable for use with ceric-perchloric acid because its potential (1·06 v.) is too low.

Some new redox indicators have been proposed and the most interesting of these are copper phthalocyanine-4-4′-4″-4‴-tetrasulfonic acid[21], and triphenylmethylarsonium chloride[22]. The former indicator appears to have some advantages over ferroin under certain conditions; it has the advantage of being cheap and very stable. Triphenylmethylarsonium chloride is used as an extraction indicator and is recommended for titrations with permanganate, dichromate and iodine. It is especially useful when the test solution is coloured with ions such as copper, cobalt, and nickel.

An interesting method for the direct cerimetric titration of oxalate with ferroin as indicator has been described by Rao and Rao[23]. This titration must normally be carried out at elevated temperatures at which ferroin is very unstable. Certain catalysts have been proposed which accelerate the reaction at lower temperatures, but none of these is very satisfactory. Rao and Rao found that sulphate ions had an inhibiting effect on the oxidation and if barium chloride is added to the solutions so that sulphate ions are removed, the oxidation is instantaneous in the cold without the need for catalysts. Unfortunately, it is necessary to work with somewhat smaller quantities than are normally used in this titration.

Complexometric Indicators

The rapid adoption of EDTA and similar complexans as titrimetric reagents has resulted in a large number of new complexometric indicators being advocated. An excellent survey of the subject is already available in the literature[24].

We have developed several of these indicators[25,26,27], and some of these studies are now described. By condensing alizarin with formaldehyde and iminodiacetic acid, 3-aminomethyl-alizarin N:N-diacetic acid was obtained[28]. This is suitable as indicator in the titration of certain heavy metals at a pH lower than can be tolerated with the usual indicators. Applications have already been found[29].

Perhaps the most interesting reaction of this compound is not as an indicator, but as a direct colorimetric reagent for fluoride[30;31] this is the first direct fluoride colour reaction to be reported in the literature. The reagent forms a red chelate with cerium(III). When fluoride ions are added, a new complex is formed which is blue; it is possible to use this reaction for the colorimetric determination of fluoride.

When H acid is diazotized, a compound is formed which gives a selective colour reaction with calcium[32]. Only nickel gives a similar reaction and this is by no means as intense as the calcium reaction. Barium and strontium are without effect. This enables calcium to be titrated in the presence of barium using CDTA in place of EDTA as titrant. The titration of calcium in the presence of strontium is not so satisfactory because of the proximity of the complex constants. The structure of the product is still unknown but work on this subject is now in progress.

For several years we have studied benzidine and related compounds as analytical reagents. More recently we have introduced the complexan type of grouping into benzidine compounds and this has yielded some new interesting indicators[33]. The following compounds have been made: *o*-dianisidine-N:N′:N″:N‴-tetra-acetic acid(I), *o*-phenetidine-N:N′N″-N‴-tetra-acetic acid(II) and benzidine-3-3′-dicarboxylic acid-N:N′:N″:N‴-tetra-acetic acid. These compounds fluoresce strongly in U.V. light and (I) and (II) have the property of forming complexes with copper and mercury which are non-fluorescent. Both these metals can therefore be titrated with EDTA using (I) or (II) as the indicator. The solution fluoresces vividly in U.V. light at the end-points which are very sharp and are much superior to those given with other indicators available for this titration. (III) behaves similarly with calcium. Further studies on this type of indicator are now in progress.

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Dr. J. E. Salmon delivers his lecture under the chairmanship of Prof. D. P. Craig.

ION EXCHANGE TECHNIQUES

by J. E. Salmon

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Battersea College of Technology

Introduction

It is now just 25 years since Adams and Holmes reported the discovery of ion-exchange resins and it would seem appropriate at this stage to take stock of the uses which have been found for these materials in the first quarter century. Some of these uses had been anticipated in the applications of natural and synthetic zeolites and sulphonated coal exchangers, but, apart from the one instance of water softening, on a very limited scale only.

The phenol-formaldehyde resins devised by Adams and Holmes have been replaced for many purposes by the more stable exchangers based on cross-linked polystyrene (or similar structures, such as cross-linked polymethacrylic acid). Furthermore additional ion-exchange materials have been introduced for specific purposes. These include phosphorylated cellulose for use in ion exchange paper chromatography, liquid ion-exchangers for applications of a solvent extraction type, ion exchange membranes for electrodialysis, and synthetic inorganic ion-exchangers for use at high temperatures or at high levels of radioactivity. The range of materials available has increased markedly in the last ten years and as a consequence new techniques based on such materials have been developed.

Ion-exchangers whatever their type are either acids (if cation exchangers) or bases (if anion exchangers) or salts of those acids or bases, but whether in acidic, basic, or salt form they are always insoluble in a wide range of solvents, including water. Fig. 1 illustrates schematically the presumed detail of a typical strongly acidic cation-exchanger in its acid or hydrogen form. In this structure the hydrogen ions, which in fact are solvated, are mobile and may be replaced by an equivalent amount of another cation

$$m\overline{H^+} + M^{m+} \rightleftharpoons mH^+ + \overline{M^{m+}} \qquad (1)$$

(where barred formulae indicate ions in the resin phase and 'm' the valency of M), while the sulphonic acid anions are bound by stable covalent bonds to the polymer network and cannot be exchanged. For solutions of specified volume and initial salt concentration and for a specified weight of resin an equilibrium will be established (equation 1) and is governed essentially by a Donnan type relation, but modified by the effect of swelling pressure changes in the resin as one cation is replaced by the other.

The strongly basic anion-exchanger shown in Fig. 2 is in its chloride (salt) form, but the free base form is obtained from this if a suitable excess of sodium hydroxide solution is passed through a column of the exchanger. In this way the equilibrium

$$\overline{Cl^-} + OH^- \rightleftharpoons Cl^- + \overline{OH^-} \qquad (2)$$

is displaced completely from left to right. The other salt forms of the anion exchanger are readily prepared from the hydroxide (free base) form.

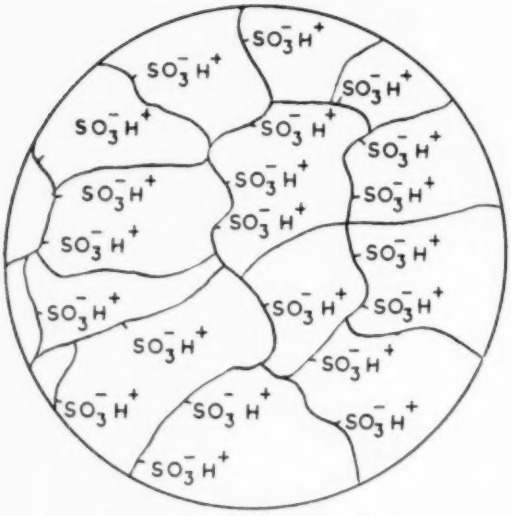

Fig. 1. Cation exchanger (schematic).

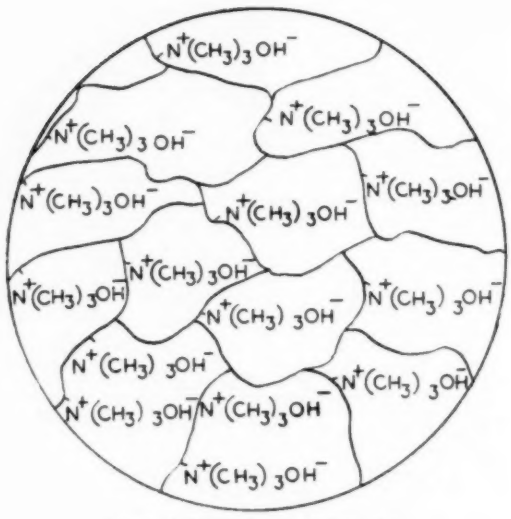

Fig. 2. Anion exchanger (schematic).

Applications in Trace Analysis

It is perhaps one of the major advantages of the ion-exchange process that the reactions involved occur readily even if the concentration of the ion initially present in the aqueous phase is only 10^{-9}M (and probably even lower). Thus a suspension of an 'insoluble' salt, such as cobalt phosphate, will contain a minute amount of dissolved salt, yielding a low concentration of cobalt ions which exchange with hydrogen ions from a cation exchanger. With a large excess of exchanger the insoluble phosphate will quickly 'dissolve' if the suspension is agitated, forming a solution of phosphoric acid and leaving the resin finally in a mixed hydrogen plus cobalt form.

$$\overline{Co_3(PO_4)_2.xH_2O} + \text{excess } \overline{H^+} \rightarrow 2H_3PO_4 + \overline{3Co^{2+}} + (\text{excess}) \overline{H^+} \quad (3)$$

Advantage is taken of exchange reactions occurring at low ionic concentrations in the use of ion-exchange resins to increase the sensitivity of spot tests. For example, if ammonium thiocyanate is added to an acid solution of iron (III) which is 10^{-6} — 10^{-8}M in iron, no visible coloration is produced, but if this solution is then passed through a column of an anion-exchanger in the thiocyanate form a red coloration is clearly visible on the resin—presumably due to the sorption of an anionic thiocyanto iron (III) complex on the surface of the beads.

Ion-exchange reactions with very dilute solutions can also be used in quantitative analysis. If an ion such as copper (II) or iron (III) is present in the range 10^{-7} to 10^{-8}M it may be sorbed from a large but known volume of solution and subsequently displaced from the exchanger with a smaller, and again known, volume of acid solution. Its concentration is thus increased by a known factor and at the same time brought into the range where sensitive physico-chemical methods may be used for its determination.

In experiments of the type just described the purity of the reagents and of the water used in preparing solutions is of particular importance. Here again ion exchange can often be used to advantage. Thus water from which the gross impurities of hardness have largely been removed by distillation or by two-stage demineralization may be freed from the remaining ionic impurities by passing it through a column of mixed-bed resin, when water with a conductivity of less than 0·1 micromhos per cm. is easily produced.

As an instance of the purification of reagents it will suffice to quote the preparation of a potassium hydroxide solution that will contribute a negligible blank in the determination of silica present in water to the extent of 0·01 p.p.m. A solution of potassium sulphate (1M) is passed through a fully regenerated (OH-form) anion exchanger.

General Analytical and Preparative Applications

The removal of interfering ions and metallic impurities in analytical and preparative chemistry can be achieved both by direct exchange of the simple cations and by anion-exchange reactions with complex anions formed by the metal ions with suitable ligands. One example of each type will be quoted by way of illustration.

In the analysis of borates containing heavy metal impurities these latter can be eliminated, prior to the determination of boron, by their sorption on the hydrogen form of a cation exchanger. The exchange is a straight forward one in which the metallic cations are replaced by an equivalent of hydrogen ions.[1]

Since, in solutions of high chloride ion content, iron

(III) forms the complex anion $(FeCl_4)^-$ which is strongly sorbed by an anion exchanger which is initially in the chloride form, it is possible to remove iron (III) impurity from concentrated solutions of hydrochloric acid or of lithium, magnesium, calcium or aluminium chlorides. The other metallic ions mentioned either do not form anionic chloro complexes or at least do not form ones which are sorbed under the conditions specified.

Such specific reactions can often be of great practical importance as in the separation of uranium from ores containing as little as 0·1 per cent of that metal. The ground ore is leached with dilute sulphuric acid, when uranium and various other metals, including iron, pass into solution. Of the metals present, however, only uranium and iron (III) form sulphato complexes which are sorbed by the sulphate form of an anion exchanger. The other metals pass through the column of ion-exchanger into the effluent. The uranium and iron which are retained on the column are subsequently displaced from it by the action of acidified sodium nitrate (Fig. 3 illustrates a typical plant).

The first reports of the use of liquid ion exchangers appear to relate to the extraction of uranium.[2] The liquid anion exchangers are essentially tertiary amines with long alkyl chains. They are soluble in such solvents as kerosene or benzene, but insoluble in water and behave as weakly basic anion-exchangers—that is they behave as anion-exchangers towards acidic solutions, but revert to the free amine state when in contact with basic solutions. As a consequence they are capable of extracting the uranium and ferric sulphate complexes from the acid leach liquor by an ion-exchange reaction. Since two liquid phases are involved, however, the procedures which can be employed are essentially those of solvent extraction and it has been claimed that this offers advantages over the conventional ion exchange method. Liquid cation exchangers such as diethylhexyl-phosphoric acid have also been prepared.

In quantitative analysis use is often made of the replacement of the cation of a salt by an equivalent amount of hydrogen ions or sometimes of the replacement of the anion of the salt by an equivalent of hydroxide ion. This application of ion exchange, sometimes known as salt-splitting, is used in the determination of the total salt concentration of solutions. By the use of a column containing an excess of hydrogen form cation exchanger, the total salt content of an aliquot of a solution may be converted to an equivalent quantity of acid, e.g.

$$\overline{NaClO_4} + H^+ \longrightarrow HClO_4 + \overline{Na^+} \qquad (4)$$

which may be determined by titration with alkali. In some instances it is necessary to use the corresponding reaction with an anion exchanger, e.g.

$$\overline{NaNO_2} + OH^- \longrightarrow NaOH + \overline{NO_2^-} \qquad (5)$$

The salt splitting process offers a method for the preparation of acids or bases which is generally much more convenient than the classical procedures, e.g.

$$\overline{Na_2H_2P_2O_6} + 2H^+ \longrightarrow H_4P_2O_6 + \overline{2Na^+} \text{ (see ref. 3)} \qquad (6)$$

Such reactions take place readily because the hydrogen ion is much less strongly held by a strongly acid (e.g. sulphonic acid) exchanger than are most other cations—that is the equilibrium constant of the reaction of equation (1) is generally greater than unity. Likewise, the hydroxide ion is less readily sorbed by a strongly basic (quaternary ammonium) anion exchanger than are most other anions.

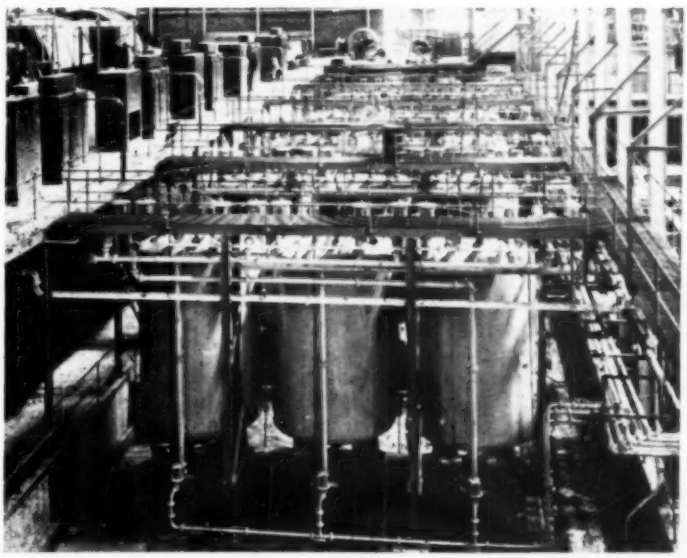

Fig. 3. Ion exchange plant for uranium extraction.

Photo courtesy The Permutit Co., Ltd.

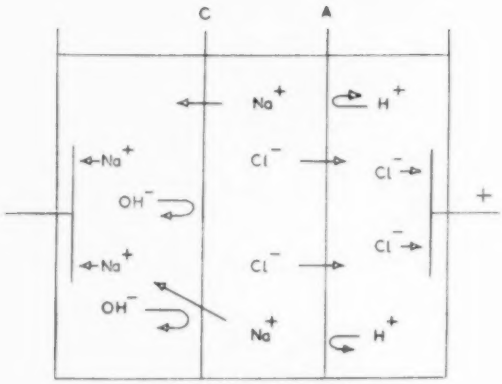

Fig. 4. Electrodialysis cell.

It is as a result of this low affinity of the hydrogen and hydroxide ions for the exchangers that a large excess of acid or base is required for the complete regeneration of cation- or anion-exchange resins respectively. This factor which is of no great importance in the analytical uses of ion exchange becomes one of economic significance in industrial applications.

Ion-Exchange Membranes

Because of the need to recover the species sorbed and to regenerate the resin, the use of ion-exchange beads in a column is of necessity a discontinuous process. Although ingenious methods have been devised to avoid this difficulty none appears to have been entirely satisfactory. The alternative is to use electrodialysis with ion exchange membranes. These are in essence thin sheets of cation-exchange or anion-exchange material and Fig. 4 illustrates a typical three compartment cell which contains one membrane of each type. The centre compartment contains sodium chloride solution, whilst a trace of sodium hydroxide and of hydrochloric acid have been added to the cathode and anode compartments respectively to give conducting solutions in all three compartments. Sodium ions migrating towards the cathode on reaching the cation-exchange membrane can be taken up by it and transported through it (effectively jumping from one sulphonic acid group to the next) so that the number of sodium ions leaving the membrane on the cathode side exactly balances the number entering on the side remote from the cathode. By contrast the hydroxide ions in the cathode compartment which move up to cation exchange membrane in their migration towards the anode cannot be taken up by the membrane and hence cannot be transported through it. Thus as a result of the effect of the permselectivity of the membrane and the cathode reaction the concentration of sodium hydroxide in the cathode compartment is increased. In a similar manner the concentration of hydrochloric acid in the anode compartment is increased by the permselectivity of the anion exchange membrane and the anode reaction. At the same time the concentration of salt in the centre compartment is reduced by the overall process which is essentially one of salt-splitting.

There is no need for a regeneration cycle for, in effect, the process of regeneration of the exchangers is being carried out continuously. Ion exchange membrane cells have been used for regenerating spent acid pickle liquors and, in the form of multicompartment cells for desalting brackish waters (Fig. 5). In practice the desalting is not complete since the electrical resistance of the compartments would become two high—if required the last traces of electrolyte are removed by conventional ion-exchange columns.

Ion-Exchange Chromatography

Ion-exchange chromatography is based on the differences in the affinities which various ions exhibit for the exchangers. Absolute values of these affinities are not known, but the relative affinities which two ions participating in an exchange reaction exhibit under specified conditions can be determined. These relative affinities are a function of both the charges on the ions and their solvated ionic radii.

Chromatographic separations based on ion exchange can be achieved using papers prepared from aminated or phosphorylated cellulose, but the majority of separations reported in the literature have been made

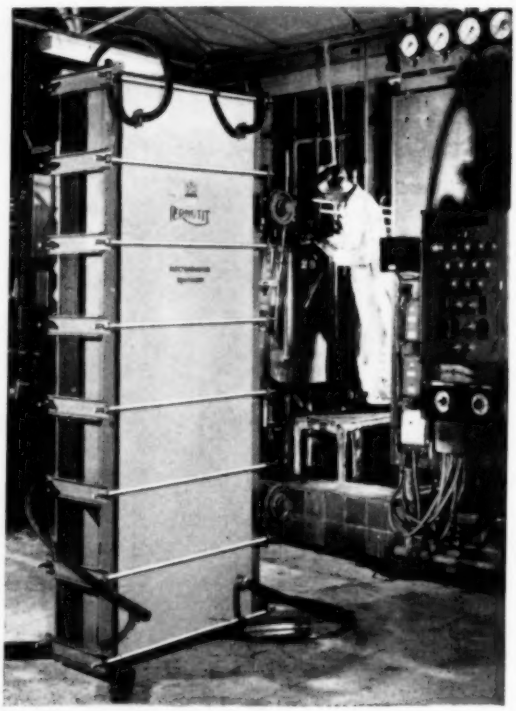

Fig. 5. Electrodialysis equipment. *Photo courtesy The Permutit Co., Ltd.*

with columns filled with conventional ion-exchange resin beads—albeit often of very small diameter. With the ion-exchange columns three chromatographic procedures may be employed, but one of these, namely elution chromatography, is commonly used for analytical purposes because it gives a greater degree of resolution of the components of a mixture.

In elution chromatography the ions to be separated are sorbed in a band at the top of a column (which is commonly in the hydrogen or ammonium forms if a cation exchanger), care being taken that only a small fraction of the total column capacity is employed. The ions in the band formed at the top of the column are then displaced down the column by the passage through it of an excess of a solution containing an ion of lower relative affinity than any of the ions forming the band (again usually hydrogen or ammonium ions for cation exchange separations). In this way the ions are displaced slowly down the column, being continuously resorbed and then displaced at each stage. During this process the components in the mixture which have the highest relative affinity for the resin are displaced more slowly down the column than those with a lower relative affinity and hence a separation is achieved.

In a series of ions of very similar chemical properties, such as the lanthanide or actinide elements, where the ionic charge is constant the principal factor determining the relative affinities is the hydrated ionic radii of the ions. The ion of largest hydrated size being the least firmly held. Hence since the ionic radii of the lanthanide ions increase steadily in size from lanthanum to lutetium in their hydrated states (in contrast to the unhydrated radii which decrease steadily in the same order), lutetium is the least firmly held of the lanthanide ions and is the one most easily displaced from the column.

With ions which are of such closely similar character, the separations obtained are more satisfactory if the effect of these differences in affinity is supplemented by that of a reaction between the ion and a complexing agent (e.g. citrate)

$$\text{e.g.}\quad M^{m+} + m\overline{NH_4^+} \rightleftharpoons \overline{mNH_4^+} + M^{m+} \qquad (7a)$$

$$\text{and}\quad M^{m+} + \text{citrate} \rightleftharpoons \text{(M-citrate) complex} \qquad (7b)$$

The effect of the second reaction (equation (7b)) will be to reduce the concentration of free metal ions in solution and hence the equilibrium of the first reaction will be displaced to favour the left to right reaction (equation (7a)). Since the smallest ion (in the unhydrated state) of a series of otherwise similar ions (i.e. lutetium in the lanthanide series) will form the complex species most readily (equation (7b)), this factor will tend to keep this ion back in the solution phase and hence make for its more rapid elution. The largest (unhydrated) ion, namely lanthanum will, however, be less affected by the complex-forming reaction and will tend to be held back on the resin. The degree of separation achieved is consequently enhanced by the complex-formation.

So regular is the sequence of separation with a given complexing agent under specified conditions that the presence of a single atom undergoing spontaneous fission in the appropriate fraction from an ion-exchange column provided the first evidence of the successful preparation of element number 101.[4] Since in these separations a high proportion of the metal present is locked up in the form of a complex ion, the total metal concentration in the solutions can be quite high, while the free metal ion concentration does not exceed the optimum value of 10^{-3}M for chromatographic separations with strongly acidic resins.

Where the reaction between the metal ions and the complexing agent leads to the formation of anoinic complexes there is then the possibility of achieving separations of metals by anion-exchange chromatography. The work of Kraus and his co-workers has provided a useful example of the exploitation of this anion-exchange procedure. Where only two ions are to be separated then the use of a complex agent that will concert one of them only to an anionic complex, will enable a simple separation to be achieved, by the use of either a cation- or an anion-exchange column, and thus the need for a chromatographic type of separation may be avoided.

In some cases complex formation cannot be used to facilitate the chromatographic separation, as is the case in the separation of the alkali metal or halide ions, or in the separation of organic cations or anions. An example of the separation of this latter class of material is provided by the very elegant procedure for the separation of amino acids by cation exchange chromatography which was devised by Moore and Stein.

One disadvantage of the method of elution chromatography is that while the first band (or elution peak, in a plot of concentration of eluted species in the effluent against volume throughput) is sharp the succeeding bands are more diffuse. Where the separation of the elution bands is great enough it is possible to change the concentration, pH, or even the chemical nature, of the eluting agent so as to speed up the elution of the succeeding bands. Such abrupt changes can cause complications, however, for example, the tailings of the preceding band may appear as a new band. A more suitable method than this stepwise elution is that of gradient elution in which the strength of the eluant is varied continuously in a predetermined manner during the elution process. As each band is leaving the bottom of the column and is being collected in the effluent, the rate of movement of the next band down the column is speeded up by the change in eluant composition. The time taken by each ion to move down the column is thus kept to the minimum consistent with the achievement of a separation. In this way sharper bands are obtained as the factors making for diffuseness in the bands are time dependent.

The versatility of the method of ion-exchange chromatography is increased by the fact that the species

to be separated need not always be ionic. For example the separation of aldehydes and ketones and of mixtures of ketones by chromatography on an anion exchange column in the bisulphite form has been reported. A similar type of reaction is involved in the uptake of iodine by the iodide form of an anion exchanger.[5] In both cases the mechanism involves the addition of a neutral molecule to an ion (already sorbed on the exchanger) to form a new ion (which is still retained by the exchanger). In the chromatographic process the bonds involved in forming the larger ion are presumably broken and re-made as the neutral species moves down the column.

Applications not Directly Dependent on the Exchange Process

In some uses of ion exchangers the exchange process is not directly involved. Such is the case in the use of ion exchangers as catalysts which depends directly on their properties as insoluble acids or bases.

One result of the Donnan-membrane type of equilibrium which prevails with ion exchange systems is that the penetration of external electrolyte—as distinct from external solvent—into the resin beads is restricted. Apart from those ions which are entering the structure to replace ions which are leaving, the ions of the surrounding electrolyte are effectively excluded from the resin beads. Neutral molecules are not so excluded and in the case of organic molecules may be absorbed (i.e. exist in a higher concentration in the beads than in the surrounding solvent) as a result of short range forces of interaction between the molecule and the resin structure. Separations of electrolytes from non electrolytes by 'ion-exclusion' have been described by Baumann and Wheaton.

Since the forces binding the neutral molecules to the resin vary from one compound to another even with the different members of a homologous series, chromatographic separations of members of such series by partition chromatography are possible.[6] A number of separations of this type using 'salting out' and 'solubilization' chromatography have been described by Rieman and his co-workers.[7]

Applications Dependent on both the Exchange Process and on the Resin Structure

It has been known for some time that certain large organic ions are unable to penetrate the structure of resins of the degree of cross-linking commonly available commercially (e.g. 8 per cent divinyl benzene (D.V.B.) for a cation exchanger) while the simple inorganic ions can penetrate highly cross-linked resins (e.g. 20 per cent D.V.B.). Use has been made of this 'ionic sieve effect' in the separation of large ions from small ions by the preferential sorption of the latter by a tightly cross-linked resin.

The active component of a number of drugs consists of a large organic ion (e.g. the alkaloids) and by the choice of a resin of a suitable degree of cross-linking it is possible to achieve a state where the large ion can only just penetrate the resin structure. In such a case the exchange process is sterically inhibited as is the subsequent release of the drug by exchange with hydrogen ions in the stomach (if the drug is cationic and sorbed on a cation exchanger) or with hydroxide in the intestine (if an anionic drug is sorbed on an anion exchanger). The result is that a slow release of drug to the system over a long period of time is achieved and the efficiency of the drug is thereby increased.

Ion Exchange in the Study of Complex-Formation

Mention has already been made of the effect of complex formation on ion-exchange equilibria (equations 7 (a) and 7 (b)). By a study of the effect of variations in the concentration of the complexing agent (with all other conditions fixed) on the sorption of tracer quantities of a metal ion, it is possible to deduce the equilibrium constant for the complex-forming reaction. The mathematical treatment has been given by Schubert and by Fronaeus.

The sorption of an acid group in the form of a group bound to a metal in a complex cation by a cation exchanger or more particularly the sorption of a metal in the form of a complex anion will also provide an indication of complex ion formation. A quantitative study of the ion exchange equilibria, by either batch-equilibrium or column studies, can yield information concerning the composition or charge of the complex ions where these are unknown. This procedure has been exploited to advantage by Kraus and his co-workers.

Where complex ions of various sizes are present as in the formation of isopolyanions by the condensation of, for example, vanadates in acid solutions, the detection of the different species present can be simplified by the use of resins of varying degrees of cross-linking. An ionic sieve effect is then superimposed on the ion exchange equilibria.[8]

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PICTORIAL SUPPLEMENT ➤

LABORATORY APPARATUS and MATERIALS EXHIBITION

JUNE 20-21-22-23 1960

Royal Horticultural Society's New Hall

Westminster, London, S.W.1

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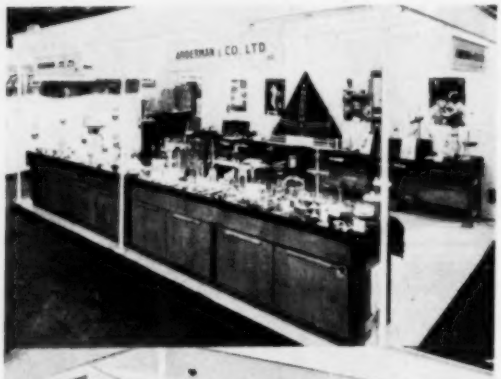
A SUCCESSFUL EXHIBITION

This new Exhibition proved to be a great success on all counts. As will be seen from this illustrated supplement, the attendance was excellent throughout and in particular was this the case at the 12 scientific lectures which were delivered to 'full houses' throughout the run of the Exhibition.

Four of these lectures are included in this issue and it is the intention to publish at least six of the remaining eight in toto *within the next three months.*

▲

Above: **After a rest in the visitors' lounge, the tour of the Exhibition is continued.**



Porcelain, glassware, chemicals, refractories and electric stirrers were featured on the Anderman stand, together with a Jena water bi-distiller which will produce four litres of bi-distilled water per hour. ▶

Astell Laboratory Service Co. Ltd. showed a selection of equipment covering the fields of chemistry, microbiology, laboratory control and laboratory furniture, with particular application to dairy and food industries. ▶

Barlow-Whitney's selection from their wide range of smaller process heating and testing equipment for laboratories drew many interested enquirers. ▶

Above we show the B.D.H. stand on which the company exhibited something of the range of its activities, including reagents, biochemicals, indicators and microscopical stains, ion exchange material and molecular sieves; the Camlab (Glass) Ltd. stand where emphasis was on thermostatically controlled laboratory equipment, including ovens and water baths; and the stand of Catalin Ltd., on which were shown examples of covalent and ionic models for use in the teaching of chemistry.

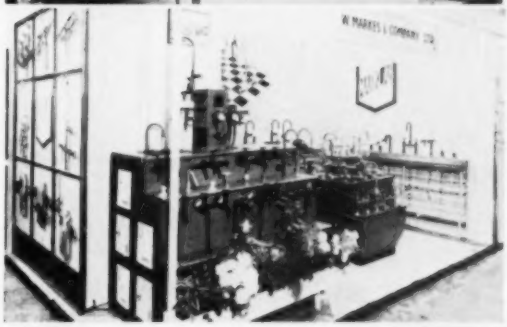
Below are the stands of Ralph Cuthbert Ltd., Endecotts (Filters) Ltd., and Esavian Ltd. On the first the company showed a wide selection of their own products together with some items of equipment manufactured by D. A. Gunn (Engineering) Ltd.; on the second the well-known range of test sieves to B.S. and U.S. Standards manufactured completely from stainless steel; and on the third examples of the Esavian Unilab units which will fit compactly into any laboratory.

Top left is the stand of W. G. Flaig & Sons Ltd., who featured a representative selection of Exelo brand laboratory glassware. *Top right* can be seen a comprehensive array of heat-controlled laboratory apparatus, furniture and fittings shown by Charles Hearson & Co. Ltd. *Left* is the Flatters & Garnett stand on which they demonstrated a range of microscopical and biological equipment with particular emphasis on microprojection.

A busy scene outside the stand of T. Gerrard & Co., Ltd. Here were to be seen some interesting examples of zoological dissections, histological, embryological, anatomical and other models, and the preparation of microscopical slides by various micro-techniques.

Above: **V. A. Howe & Co., Ltd. assembled a cross-selection of instruments of U.S. and continental manufacture, including pH equipment, fraction analysers, centrifuges and microtomes. More continental equipment was shown on the stand of the Hudes Merchandizing Corporation, and Isopad Ltd. displayed a full range of electric heating mantles from the smallest micro type to the largest pilot sizes made for flasks up to 200 litre capacity.**

Below: **The Laboratory & Electrical Engineering Company showed the latest developments in laboratory incubation, a brushing machine and needle sharpening attachment, and a mercury vapour U.V. lamp. Marklab exhibits included taps, cocks and valves, and a selection of fittings of special interest to the medical research and pathological laboratories. Laboratory shelving and benching, fume cupboards and fume extraction equipment formed part of the display staged by the North of England School Furnishing Co., Ltd.**



Below: Furnaces, water baths, centrifuges, heating mantles, balances, deionizers and a selection of laboratory and scientific glassware were featured by J. Preston, Ltd.

▼

Below: The chief exhibit featured by Silverson Machines (Sales) Ltd., was a new high speed mixer emulsifier designed especially for laboratory use.

▼

▲ *Above:* Marks 4 and 5 Demin-rolit units and the new Mark 6 model were featured by the Permutit Co., Ltd., together with samples of ion exchange resins for the laboratory.

Right: A busy moment outside the United Trade Press stand. ▶

◀ *Left:* A wide display of technical books were displayed by members of the Publishers Association.

Left: A range of general laboratory apparatus with special emphasis on their well-known 'Off the Shelf' service was featured by Scientific Supplies Co., Ltd. ◀

Right: The Worcester Royal Porcelain Co., Ltd. shared a stand with John Moncrieff Ltd., to show a wide range of laboratory porcelain and glassware. ▶

▲

Winkworth Machinery Ltd., A. D. Wood and X-Lon Products Ltd. showed respectively (*a*) mixers with demonstration models to show blade actions; (*b*) a complete range of equipment that they supply together with some of their own range of high vacuum glass stopcocks; and (*c*) plastic laboratory equipment, including beakers, measuring cylinders, pipette washers, funnels, etc.

Features of the Exhibition were the daily demonstrations of glass-blowing given by Mr. J. Frost of the Chemistry Department, University of Reading—these were always a great attraction. ▶

The daily lectures were also a great success and were in all cases (as seen below) delivered to 'full houses'. Four of these lectures appear elsewhere in this issue.

▼

STAND NUMBERS AND ADDRESSES OF EXHIBITORS

Aimer Products Limited, 56/58 Rochester Place, Camden Town, N.W.1.	7
Anderman & Company Ltd., Battlebridge House, 87/95 Tooley Street, S.E.1.	32
Astell Laboratory Service Co. Ltd., 172 Brownhill Road, Catford, S.E.6.	23
W. & R. Balston Ltd., c/o H. Reeve Angel & Co. Ltd., 9 Bridewell Place, E.C.4.	59
Barlow-Whitney Ltd., 2 Dorset Square, N.W.1.	53
Boro' Laboratories & Appliances Co. Ltd., 1 Station Buildings, Catford, S.E.6.	30
The British Drug Houses Ltd., Graham Street, N.1.	24
Camlab (Glass) Ltd., 50 Burleigh Street, Cambridge.	47
Catalin Limited, Farm Hill Road, Waltham Abbey, Essex.	41
Communication Systems Limited, c/o Automatic Telephone & Electric Co. Ltd., Publicity Dept., St. Vincent Street, Liverpool, 3.	37
Cooke, Troughton & Simms Ltd., York.	64
Alfred Cox (Surgical) Ltd., Edward Road, Coulsdon, Surrey,	27
Ralph Cuthbert Ltd., 26 Mortimer Street, W.1.	60
Cygnet Joinery Ltd., Higher Swan Lane, Bolton, Lancs.	52
F. Darton & Co. Limited, Watford Field, Watford, Herts.	29
Degenhardt & Co. Ltd., 6 Cavendish Square, W.1.	54
Electrothermal Engineering Limited, 270 Neville Road, E.7.	35
Elga Products Limited, Lane End, Bucks.	1
Elliot Brothers (London) Ltd., Century Works, Lewisham, S.E.13.	18
Endecotts (Filters) Limited, Lombard Road, S.W.19.	45
Esavian Limited, 185 Tottenham Court Road, W.1.	19
Esco (Rubber) Ltd., 34/36 Somerford Grove, N.16.	2
W. G. Flaig & Sons, Ltd., Exelo Works, Margate Road, Broadstairs, Kent.	26
Flatters & Garnett Ltd., 309 Oxford Road, Manchester, 13.	4
T. Gerrard & Co. Ltd., 46 & 48 Pentonville Road, N.1.	34
Grant Instruments (Cambridge) Ltd. Barrington, Cambridge.	56
Geo. M. Hammer & Co. Ltd., Crown Works, Hermitage Road, N.4.	66
Charles Hearson & Co. Ltd., 68 Willow Walk, S.E.1.	21
Hendrey Relays Limited, 392 Bath Road, Slough, Bucks.	42
F. J. Hone & Co. Ltd., 19 Eldon Park, S.E.25.	17
V. A. Howe & Co. Ltd., 46 Pembridge Road, W.11.	48
Hudes Merchandising Corporation Limited, 52 Gloucester Place, W.1.	57
Isopad Limited, Barnet By-pass, Boreham Wood, Herts.	15
Laboratory & Electrical Engineering Co., Goldsmith Street, Nottingham.	31
Laboratory Equipment (London) Ltd., Libra Works, 18-20 Dames Road, E.7.	20
L. Light & Company Limited, Poyle, Colnbrook, Bucks.	50
W. Markes & Company Ltd., Wolverhampton Road, Wedges Mills, Cannock, Staffs.	14
May & Baker Ltd., Dagenham, Essex.	36
National Research Development Corporation, 1 Tilney Street, W.1.	51
The North of England School Furnishing Co. Ltd., East Mount Road, Darlington.	43
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Prof. R. W. Douglas lectures on glass as a laboratory material (Chairman: Prof. S. Tolansky.)

GLASS

IN THE LABORATORY

by

R. W. Douglas

Professor of Glass Technology, Sheffield University

IT IS difficult to think of a laboratory in which glass is not used in one or other of its forms. Different laboratories have different uses for glass; in the chemical laboratory most of the apparatus is made from one type of glass, the so-called heat resisting borosilicate glass, other glass articles such as desiccators, windows and bottles will be of a soda-lime-silica glass, made from sodium carbonate, calcium carbonate and sand with a few minor constituents.

The borosilicate glass has a lower coefficient of expansion than that of the common soda-lime-silica glass and it is to this that it owes its greater resistance to a sudden change in temperature. But there are, of course, many other glass compositions which have their special application in the laboratory and in industry; by varying the chemical composition of the glass it is possible to select, within limits, glasses which have particularly useful physical properties for various applications. For example, glasses containing a large amount of lead oxide are used for windows, which at the same time as being transparent, absorb high energy rays and particles. Other glasses may be chosen because they have the ability of resisting the attack of alkaline vapours as, for example, the special glass used in the interior of the sodium vapour street lighting lamps. In the optical glass field many interesting new developments are taking place using as constituents the more uncommon elements such as, for example, lanthanum and germanium. In view of the great variety of apparatus which employs glass it is impossible in a brief review to do more than touch upon a few topics; the emphasis in this contribution has therefore been placed much more on glass in the glass technologist's laboratory than on details of the use of glass in other laboratories. The account which follows gives a short introduction to the material glass, the vast range of compositions which can be employed and at the same time, a few glimpses of the physical and chemical problems which face a student of glass.

All these materials have one thing in common: they have been prepared by the fusing together of a mixture of inorganic oxides which has subsequently been cooled to room temperature where it has the mechanical properties of a solid and has changed from a liquid to a solid without precipitating crystals.

The Formation of Glasses

When a normal liquid which does not form a glass is cooled, there is an instantaneous change of state at the freezing point usually accompanied by the discontinuous change in volume. Thereafter, the coefficient of expansion is smaller than it was in the liquid state. If a liquid is to form a glass it must be cooled through this critical temperature sufficiently quickly to prevent the formation of crystals. The rate of formation of crystals will depend upon the number of nuclei present and the rate of growth of the crystal is roughly related to the viscosity. All the glasses met with in the laboratory have high viscosities at the freezing point or liquidus temperature.

The term 'liquidus temperature' must be used instead of 'freezing point' when dealing with a glass of complex composition, for the crystals which separate from the liquid will not, in general, have the same composition as that of the liquid; the liquidus temperature being defined as the highest temperature at which these

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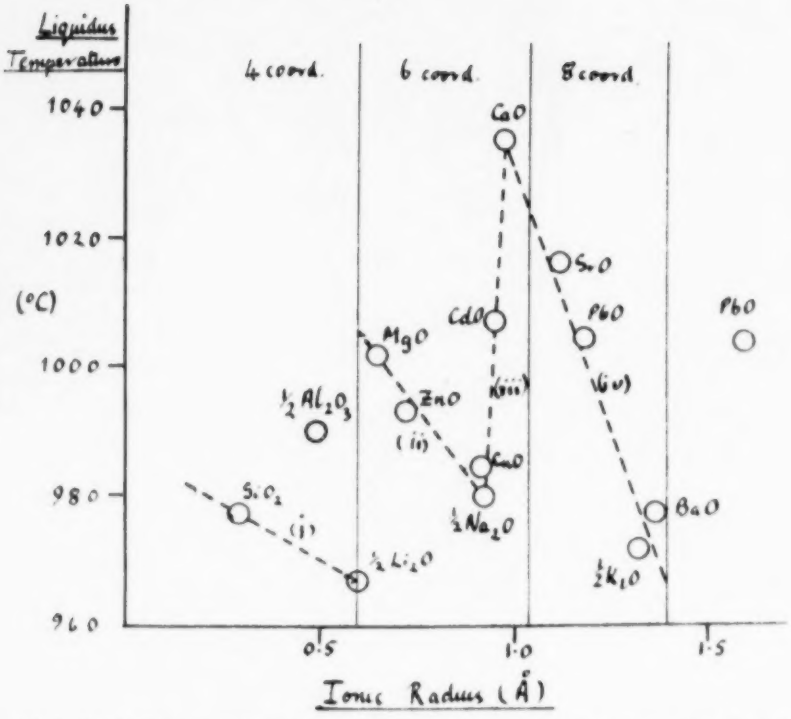

Fig. 1. (S. P. Jones and R. W. Douglas. To be published, Physics and Chemistry of Glasses, 1960, No. 6). Effect on the liquidus temperature of the substitution of one mole per cent of CaO in a soda-lime-silica glass by various oxides.

crystals can exist in contact with the liquid. For example, when sodium oxide is added to silica the liquidus temperature falls rapidly and when 30 per cent of sodium oxide has been added the liquidus temperature is about 800°C. This material forms a glass which is fairly rapidly soluble in water, and in solution it is known as water glass. The substitution of lime for some of the sodium oxide improves the resistance of the glass to attack by water without raising the liquidus temperature too much. In this way the normal soda-lime-silica glass composition of commerce which is about 75 per cent silica, 15 per cent soda and 10 per cent lime is obtained.

In practice, the increased efficiency of melting furnaces has enabled the amount of soda to be reduced and several minor constituents are added to improve the quality of glass. For example, the addition of a small amount of alumina improves the resistance to attack by water and the addition of a small amount of magnesia lowers the liquidus temperature and makes devitrification less likely to occur during the forming of articles from the glass. The addition of a small amount of various oxides to a soda-lime-silica glass has recently been studied in Sheffield with interesting results. It is found that the amount by which the liquidus temperature is lowered by any given oxide depends upon the size of the metallic ion involved, and also upon the number of oxygen ions with which it is normally surrounded in crystals (Fig. 1). This behaviour leads to the conclusion that each of these cations when added to glass surrounds itself by a fixed number of silicon-oxygen groups.

These results are obtained when the amount of added oxide is sufficiently small not to alter the type of crystal precipitated. It is possible to add to glasses materials which are themselves precipitated more easily than the primary crystalline phase of the parent glass. For example, if a little of a mixture of cadmium sulphide and cadmium selenide is added to the glass mixed crystals of these elements can be caused to precipitate by heat treatment at a lower temperature than the normal liquidus temperature. When precipitated these crystals give orange and ruby colours to the glass according to the ratio of sulphur to selenium.

The recently developed Pyro-Ceram materials can be considered as an extension of these two processes in combination. By addition to an appropriate glass mixture of a nucleating agent the primary phase can be caused to grow on nuclei provided by the foreign nucleus, and if the composition of the glass is suitably chosen, nearly the whole of the material can be converted from glass to crystal by appropriate heat treatment. Thus, it is possible to fabricate articles by the normal methods of glass manufacture and then to convert the glass article to a ceramic by heat treatment; however, some of the glasses used in this process are so liquid at high temperatures that special means of fabrication have to be adopted.

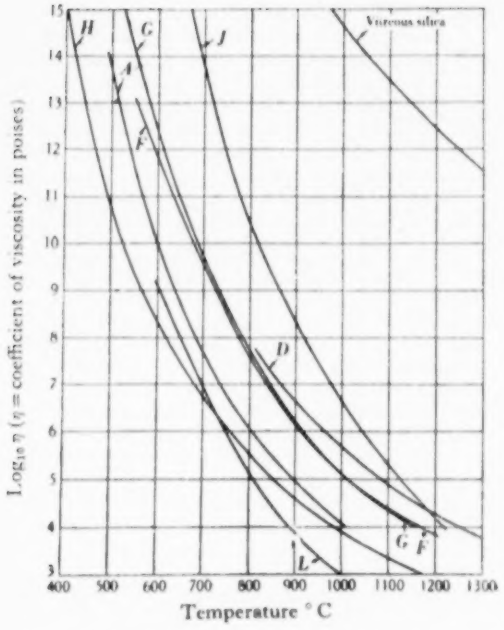

Fig. 2. Relation between viscosity and temperature for various glasses. (A) soda-lime-silica glass (D) laboratory borosilicate glass (G) and (F) tungsten sealing borosilicate glasses (J) mercury vapour resistant glass (H) lead crystal glass (L) sodium vapour resistant glass.

Viscosity

Assuming that the composition of the mixture is appropriately adjusted so that devitrification is avoided, a glass forming liquid then continues to cool as a supercooled liquid. Below the liquidus temperature its viscosity increases rapidly as the temperature falls until the viscosity has become so high that the glass will have the normal properties of a solid as defined in the elementary way by saying that a solid is a substance which retains its shape.

Commercial glasses are in general made by heating the batch mixture in a furnace at temperatures between 1,400°C. and 1,500°C. After the reaction is complete and the bubbles have disappeared the temperature of the glass is lowered somewhat so that it is in the right condition for gathering from the furnace for fabrication either in a machine or by hand. In hand-working, the glass may be made in a pot and the adjustment of the temperature done in a time sequence, whereas, when large automatic machinery is used for fabrication, the glass is melted in a tank furnace and the adjustment of the temperature is done in space, i.e. as the glass flows from the melting end through the furnace to the working-out end the temperature is controlled so that it varies suitably along the tank to provide glass at the right temperature for the machines. As the glass cools its viscosity increases rapidly and the art of fabrication of a glass article depends therefore upon shaping the article and removing the heat from it at the right rate so that by the time the processing is finished the article is just set sufficiently solid to retain its shape, i.e. the viscosity of the outer layers is sufficiently high. The

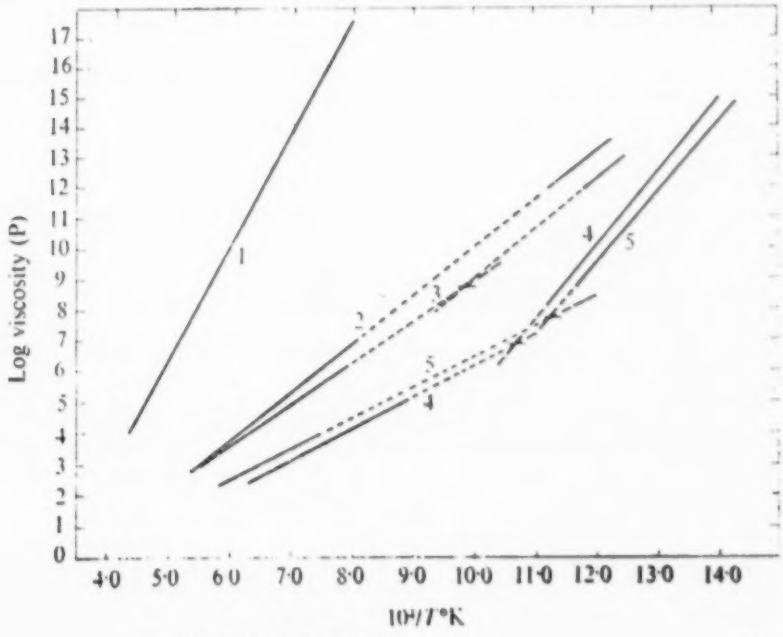

Fig. 3. (After C. R. Kurkjian and R. W. Douglas Physics and Chemistry of Glasses, vol. No. 1 page 19, 1960). Viscosity temperature relationship: (1) SiO_2; (2) GeO_2; (3) 0·045Mol% Na_2O in GeO_2; (4) 20Mol% Na_2O in SiO_2: 24·5 Mol% K_2O in SiO_2.

Note: The intermediate sections of curves 3, 4 and 5 are approximate only.

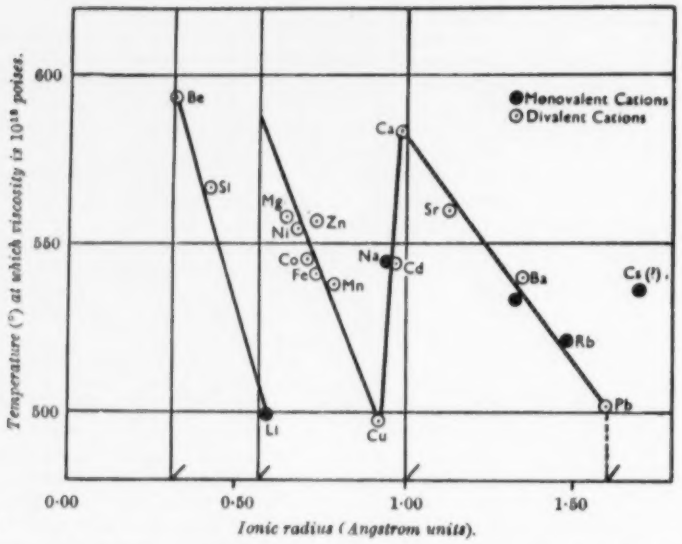

Fig. 4. (After A. G. F. Dingwall and H. Moore, J.S.G.T., 37 316, 1953). Effect of the substitution of 8 per cent SiO_2 in a soda-lime-silica glass by various oxides, on the temperature at which the viscosity is 10_{12} poises.

article must then be cooled slowly, that is, annealed, to prevent its spontaneous fracture when it reaches room temperature.

Examples of the way in which the viscosity of glass varies with temperature are given in Fig. 2. It should be noted that the scale of viscosity is logarithmic; some idea of the implication of this scale can be given by imagining that we could measure the viscosity at a temperature where it is equal to 100 poises, that is, ten thousand times greater than the viscosity of water at room temperature, by observing the rate of fall of a sphere of steel of 1 mm. diameter through the glass. This rate would turn out to be about 1 cm. per second. If we now could cool this glass until its viscosity was 10^{14} poises then the rate of fall of the sphere would become 1 cm. in 10^{12} seconds, which is 1 cm. in about one million years.

The enormous variation of the viscosity with temperature is a very interesting phenomenon which is not yet fully understood. Quite recently it has been established that the logarithm of the viscosity of the simple glasses SiO_2 and GeO_2 varies linearly with the reciprocal of the absolute temperature, but immediately the small addition of an oxide such as Na_2O is made, the plot is no longer linear but can roughly be described by two straight lines (Fig. 3). This is true also of more complex glasses. It is interesting to note that the effect of the various oxides on the viscosity of a soda-lime-silica glass is quite different when the viscosity is low from when it is high (Figs. 4 and 5). This of course enables some adjustment to be made to the shape of a viscosity temperature curve by the addition of small amounts of these modifying oxides. In addition to the oxides already mentioned, boric oxide and fluorine particularly are added to lower the viscosity at high temperatures and to increase the rate of melting.

Forming of Glass Articles

The way in which the viscosity changes with time is not, however, the sole physical factor governing the forming of glass articles. It is obvious that the rate at which heat is removed is also of first importance. At temperatures

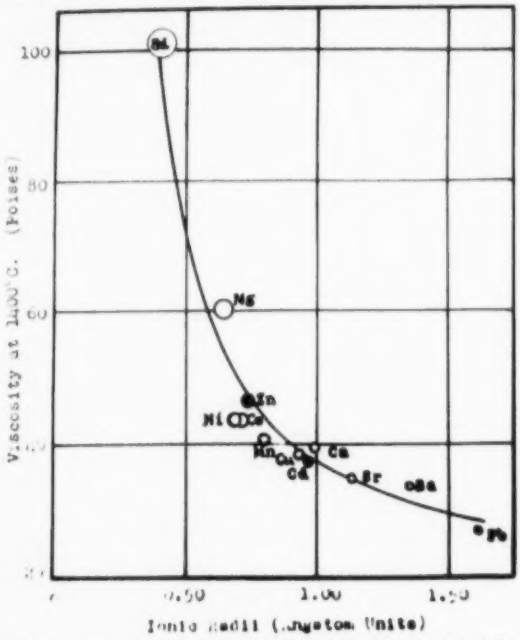

Fig. 5. (After A. G. F. Dingwall and H. Moore, J.S.G.T., 37, 316, 1953). Effect of the substitution of 8 per cent SiO_2 in a soda-lime-silica glass by various oxides at which the viscosity is 1400°.

above 1,000°C. the transmission of heat through glass becomes a complex phenomenon because there is a contribution to the apparent thermal conductivity which arises from the absorption and re-emission of radiation within the glass itself. The exact relationship between the energy which flows through the glass by this mechanism and the temperature involves the spectral distribution of the energy emitted by the glass and the absorption of the glass as a function of wavelength. A simple theory which makes approximations to avoid the need for the knowledge of the spectral energy distributions, leads to an approximate formula which shows that the conductivity varies as the cube of the temperature and inversely as the absorption coefficient. Now, it is known that small amounts of iron in the glass can cause absorption of the near infra-red radiation and variations in the amount of this iron and its state of oxidation can cause important differences in the effective thermal conductivity of glass. Iron is an ever present impurity in the raw materials normally used in glass manufacture. Even when the iron content is reduced below 0·1 per cent this residual amount still imparts an undesirable colour to the glass. Fortunately, by ensuring that as much of the iron as possible is oxidized, as for example, by the addition of oxidizing agents to the batch, the colour caused by the iron can be considerably reduced and the residual colour so obtained can be neutralized by the addition of a slight pink colour obtained by adding a little selenium. This is the process known as decolorizing.

Annealing

On cooling below the temperature at which the viscosity is above 10^{14} poises, the glass article will have a rigid exterior with the inside somewhat hotter because of the temperature gradient which exists in the cooling object. When the article reaches room temperature the inside parts will have contracted through a larger range of temperature than the outside after the outside had first become rigid and the temperature gradient, which formerly existed, will now be replaced by a distribution of stress. The inside having contracted more, will be pulling the outside inwards and so there will be compressive stress on the outside and tensile stresses on the inside. This phenomenon is probably familiar to many in Rupert Drops and is taken advantage of in the manufacture of toughened glass where the cooling is made much more rapid by vigorously blowing air on to the outside of the glass sheet which then puts the outer layers into very strong compression and, as glass always fails in tension, the strong compressive stresses have to be neutralized and additional tensile stress added to cause the sheet to fracture. These toughened plates are thus much stronger than ordinary untreated plates. Usually, however, it is desirable to remove the stresses as far as possible and this must be done by cooling slowly so that the temperature gradient in the body is kept as small as possible and thus the resulting eventual stress gradient is kept small. This is the process known as annealing.

The Strength of Glass

Even well annealed glass is, as we all know, brittle. This is a subject which is receiving much study at the present time. There are various ways of estimating the theoretical strength of glass which all lead to estimates of the order of one million pounds per square inch, whereas the strength measured on massive glass is of the order of forty thousand pounds per square inch. This large discrepancy between the practical and theoretical strength is usually attributable to the presence of microscopic flaws on the surface which act as stress magnifiers. The nature of these so-called Griffith flaws is still a mystery. There is no doubt of the very great importance of the state of the surface of glass on its strength. By careful manufacture of glass fibres strengths of the order of five hundred thousand pounds per square inch can be realized. Even in thick articles strengths of the order of two hundred thousand pounds per square inch can be obtained by treatment with certain etching solutions. After removal of about 1/20 mm. in this way very strong articles can be obtained. There is as yet, no way in which such strengthening treatments have been put to practical uses. One great difficulty is that if such strong surfaces are allowed to touch one another their strength is reduced to the original normal value and so at the present time about twenty thousand pounds per square inch must be accepted as the practical strength of glass.

Thermal Endurance of Glass

In the laboratory glass is subjected to thermal shock and the most dangerous thermal shock is that in which the outside of a hot object is cooled by plunging into the cold liquid. Here the circumstances are quite different from those which were considered when discussing the problem of annealing because now all the glass is in the rigid condition the whole time. The stresses set up will therefore be temporary ones which arise from the fact that when the temperature gradient is established in the glass, as by cooling rapidly the outside, the outside layers wish to contract and are prevented from doing so by the rigid inside, thus causing tensile stresses, dangerous ones, on the outside.

The magnitude of these stresses clearly depends upon the amount of contraction which accompanies the change in temperature and it is not surprising therefore, that there is a rough correlation between the reciprocal of the coefficient of expansion and the temperature difference which standard glass articles will withstand. This is made use of in practice in borosilicate heat-resisting glasses which have a coefficient of expansion of about one-third of the normal soda-lime-silica glass and are thus much better able to withstand the sudden changes in temperature. Ideally, fused silica would be the best heat resisting glass, but its high melting point and the difficulty of fabricating apparatus from it makes its cost prohibitive for general use. The commercial glasses can be regarded as resulting from the addition of fluxes of one kind or another to the silica in order to make large scale production at a reasonable

cost possible. It is easy to lower the melting point by many hundreds of degrees by the addition of sodium oxide. Adding too much sodium oxide will give a glass with a high coefficient of expansion and a low resistance to thermal shock; it will also lower the resistance to attack by water and other reagents, a complex term often described collectively in the title 'chemical durability'. By adding lime the resistance to chemical attack is much reduced. These two questions of reducing the melting point as far as possible and at the same time keeping the glass reasonably durable lead inevitably in the soda-lime-silica glass to compositions about 75 silica, 10 lime and 15 soda. A composition which certainly was known to the Romans and which has been modified only in the last 70 years or so, chiefly, by the reduction of the sodium oxide content still further as the efficiency of furnaces increased, and also by the addition of minor ingredients which have desirable properties as, for example, decreasing the likelihood of devitrification and changing somewhat the shape of the viscosity temperature curve.

Durability of Glass

It is customary to talk about the resistance of a glass to attack by water and other reagents under the general heading 'The Chemical Durability of a Glass', but this heading really covers a very complex set of phenomena; different glasses reacting to different reagents in different ways. A glass which is particularly resistant to acid compared with other glasses may turn out to be one of the least resistant to alkalis. With laboratory borosilicate glasses problems of durability appear to be rare but in the same system of glasses it is possible to move into a region of composition where a very pronounced reaction will occur with water or hydrochloric acid. Glass in this range of composition when given suitable heat treatment appears to separate on a micro-scale into two phases consisting of silica and a mixture of Na_2O and B_2O_3. After this heat treatment and separation of the phases it is possible by treatment with hydrochloric acid to remove the Na_2O and B_2O_3 leaving behind a skeleton consisting almost entirely of silica. A further heat treatment will cause this skeleton to contract and eventually a clear article about two-thirds the size of the original article can be obtained which contains approximately 96 per cent silica, 0·5 Na_2O and the remainder B_2O_3. This is a commercial process which provides a way of producing very low expansion glass articles which are intermediate in cost between that of the borosilicate glass and pure fused silica, the ware so produced is called 'Vycor'.

In the soda-lime-silica system the first stages of reaction with water appear to be an ionic exchange whereby ions from the water exchange places with sodium ions in the glass, and to a rough approximation the durability of such glasses varies with the sodium oxide content. The situation is, however, more complicated than this; the ion exchange reaction which involves the diffusion of sodium ions out of the glass is controlled by the rate of diffusion of these ions or the rate of diffusion of the replacing ions from the water, and this being so, the amount of reaction varies with the square root of time. However, if the reaction is continued for a long time this regime breaks down and the rate of reaction accelerates and becomes proportional to the time. In potassium oxide-lime-silica glass the second reaction takes over very quickly and indeed the diffusion controlled reaction has not been clearly isolated from the reaction which varies linearly with time.

These are new results which are quoted to emphasize the complexity of this problem of durability. Fortunately such problems rarely arise today in practice owing to the decrease of the sodium oxide content of soda-lime-silica glasses and the inertness of the borosilicate glasses. If such problems do arise in practice it is often necessary

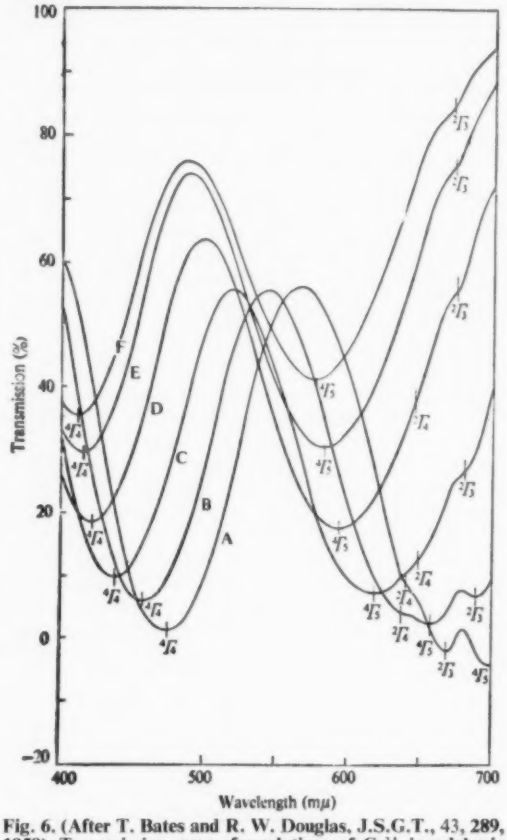

Fig. 6. (After T. Bates and R. W. Douglas, J.S.G.T., 43, 289, 1959). Transmission curves for solutions of Cr^{3+} in sulphuric acid water mixtures.

Curve	Cr^{3+} concentration	$\%H_2O$	$\%$conc. H_2SO_4	Displacement of curve
A	0·00330 M	0	100	25%
B	0·00330 M	20	80	20%
C	0·00330 M	40	60	15%
D	0·00330 M	60	40	10%
E	0·00330 M	80	20	5%
F	0·00330 M	100	0	0

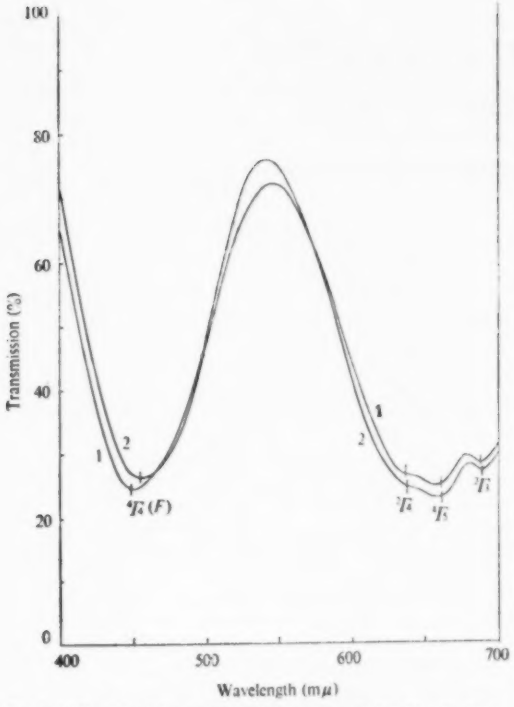

Fig. 7. (After T. Bates and R. W. Douglas, J.S.G.T., 43, 289, 1959). Comparison of the absorption curves of Cr^{3+} in a soda-lime-silica glass and an 80 per cent sulphuric acid 20 per cent water solution.

to tackle the problem as one particular to the process involved.

Optical Glasses

It was the requirements of the lens designer which initiated the development of new glass compositions which has proceeded during the last 70 years and which gave the impetus to the study of the properties of glasses as a function of their chemical composition. These developments began with the work of Abbe and Schott at Jena. At that time only two glasses were available to the lens designer—the soda-lime-silica glass (crown) and a glass containing about 32 per cent lead, 13 per cent potash and 55 per cent silica (flint) which is familiar in the home as the glass from which high quality tableware is made. Because of its high content of lead this glass has a high refractive index which gives it its lively sparkle, especially when cut into numerous facets. In order to make an achromatic doublet it is necessary that the refractive indices of the glasses used shall vary in different ways with wavelengths, i.e. that they shall have a different dispersion which is defined as $\frac{{}^{n}F - {}^{n}C}{{}^{n}D - 1}$ where ${}^{n}F$, C, D are the refractive indices for the F, C and D lines of the solar spectrum. For a long time it was found that the dispersion varied uniformerly with the refractive index and it is only in recent years that new glasses have been developed in which the dispersion is related in a different way with the refractive index. These glasses contain interesting new components such as the oxides of lanthanum and thorium.

In another field a demand has grown up for glasses which are transparent to infra-red radiation. There are two problems here. One is that all glasses contain a small amount of water which has strong absorption bands in the near infra-red. The water can be removed by special treatment, but the wavelength on which infra-red absorption begins is still around four microns for normal glasses.

Glasses in which most of the silica has been replaced by calcium oxide and alumina enable this limit to be pushed out another micron or so, and the best oxide glass is one which contains no silica but instead contained large amounts of tellurim dioxide. The absorptions which occur in the infra-red correspond to the natural frequency of vibrations of the atoms under the interatomic forces and it is inevitable that as the transmission limit in a given glass forming system is pushed further out the viscosity of the glass is reduced at any given temperature. The best infra-red transmission glasses are based on arsenic trisulphide and although these glasses transmit to about 12μ their annealing point is 150° as compared with 550° for normal soda-lime-silica glasses.

Colours of Glasses

The two methods of obtaining coloured glasses have been mentioned already. The first of these comprises the so-called precipitation colours which are obtained by arranging the composition and heat treatment so that small quantities of coloured crystals are precipitated in the glass. The cadmium-selenium ruby is an example of a precipitation colour. The other type of colours is that of the solution colours which are strictly analogous to the colours found in aqueous solutions. As is well known, ions of the elements from vanadium to copper all give coloured salts or solutions. These are the ions which have an incomplete d-shell of electrons, and the colours are due to the movement of electrons between the available levels in these d-shells. These levels are modified by the presence of the solvent ions as can be seen for example when the Cr^{3+} ion is dissolved in solutions of water and sulphuric acid. As the sulphuric acid is added to the water the absorption bands move progressively to the red end of the spectrum (Fig. 6). It is possible by taking a solution of about 80 per cent sulphuric acid to match very closely indeed the absorption curve of the same ions in the soda-lime-silica glasses (Fig. 7). This is a particularly interesting observation because these absorption curves can be discussed theoretically in terms of the ligand field theory. This leads to the conclusion that in a glass the Cr^{3+} ion is surrounded by six oxygen atoms.

COMPLEXOMETRIC ANALYSIS

by J. A. R. Genge, M.Sc., A.R.I.C.

Battersea College of Technology

Part II—DIRECT AND INDIRECT TITRATIONS

IN PART I, an outline was given of the theoretical basis of complexometric analysis and of the scope and limitations of the method. No practical details were included however and it is proposed, in this article, to give the practical procedures for the direct and indirect titrations of metal ions with ethylenediaminetetraacetic acid (E.D.T.A.). The ease with which such titrations can be carried out varies enormously with the metal itself, with the other ions present in the solution and with the physical conditions and it is hoped to give some idea of the difficulties to be expected with any given titration and, where possible, of the methods available for overcoming these.

Although the free acid, which is not hygroscopic and has no water of crystallization is, in some ways, a better primary standard, the more soluble disodium salt ($Na_2Y.2H_2O$ Mol. Wt. = 372·2) is more convenient. Its use as a primary standard has been discussed by Blaedel and Knight (1954). It is obtainable as a pure material but has to be dried at 70-80° C. because above this temperature it begins to lose water of crystallization. Alternatively the E.D.T.A. solution may be standardized with a metal, zinc being very suitable. It is important to use pure water (preferably deionized or redistilled) and the standard solutions should be stored in hard glass or, better, polythene bottles. With precautions, these solutions can be stored indefinitely.

As an example of the direct titration technique, details are given in Experiment 1 for the estimation of zinc at pH 10 using Eriochrome Black T indicator.

Experiment 1

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Buffer solution for pH 10-7 g. ammonium chloride dissolved in 57 ml. 0·880 ammonia and made up to 100 ml. with water.

Eriochrome Black T solution—0·05 g. of the solid indicator dissolved in 25 ml. water made alkaline with a few drops of ammonia.

Procedure

Ammonia is added to an aliquot of the zinc solution until it is alkaline using methyl red indicator. The final pale yellow colour does not interfere with the colour end-point of the titration. Five ml. of the buffer solution is added and followed by a few drops of the Eriochrome indicator, sufficient to give a distinct but pale wine red coloration. The titration is then carried out with the standard E.D.T.A. solution until the colour changes to a pure blue. If the conditions are correct, this colour change is very sharp.

It is very important that only pure analytical grade reagents and good quality water are used at all stages in the titration, particularly if very dilute solutions are being used. The above experiment uses 0·01 M solutions but there is, however, no reason why these titrations cannot be carried with more concentrated solutions, for example 0·1 M. The calculation offers no difficulties. The E.D.T.A. forms a 1:1 complex with all metals and one mole of reagent is equivalent to one mole of metal.

The simple direct titration technique may be used with a wide range of metals but there are some important restrictions. While most polyvalent metals form complexes with E.D.T.A., not all of them are formed rapidly enough for a direct titration. Taube (1953) has shown how the rate of complex formation and the stability depends on the electronic configuration of the metal. With metals which do not form complexes instantly, it is best usually to carry out an indirect titration but sometimes the difficulty may be overcome by titrating at a higher temperature or in a partially ethanolic solution.

The other main restriction to the use of direct titrations is that the metal must be soluble at the required pH. In general, the stability of complexes increases with an increase in pH and many metals, particularly the divalent ones, only form complexes and can be titrated only under alkaline conditions. Some of these, for example the alkaline earth metals, are soluble but many others precipitate hydroxides. Sometimes the metal can be held in solution as a weak complex as in the case of copper which can be dissolved easily as the ammonium complex. When this is not possible an indirect titration has to be used.

The subject of metal indicators in these titrations has been reviewed by Barnard, Broad and Flashka (1956 and 1957). A wide range is available commercially and new ones are being suggested frequently, the trend being to develop new indicators which will give a sharp end-point with a few or even only one metal. Lists of indicators will be found in the bibliography quoted in Part I and in chemical catalogues. While many, such as the Eriochromes and methylthymol blue are used at pH 10 or higher, some such as PAN (1-2-pyridylazo-2-naphthol) and derivatives and dithizone are used over the pH range 4 to 6 and yet others, such as pyrocatechol violet are used at pH 2 to 3. Thus the use of another indicator often provides the answer to the difficulty of keeping the metal in solution at high pH but, as might

be expected, titrations at lower pH values are applicable only to ter- and tetra-valent metals.

Almost all of these compounds are also pH colour indicators and sometimes advantage can be taken of this when adjusting the pH of the solution. This technique is used in Experiment 2, which describes the estimation of bismuth by a direct titration at pH 2·5 using pyrocatechol violet indicator.

Experiment 2

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Pyrocatechol Violet solution—0·1 g. of the solid indicator dissolved in 100 ml. water.

Procedure

A few drops of the indicator solution are added to an aliquot of the bismuth solution and the acidity adjusted with dilute nitric acid or ammonia to pH 2 to 3. The correct pH is indicated by the appearance of a blue colour. If, as is most likely in this case, the acidity is too high initially, the solution will be violet. The titration is then carried out with the standardized E.D.T.A. until the colour changes to yellow. The end-point is very sharp and is preceded by a transient purple colour.

Experiments 1 and 2 are typical of direct titrations and the procedure is much the same in all the other cases, the difference being in the indicator and hence in the pH and in the corresponding buffer solution. All determinations which can be carried out by a direct titration can also be done by an indirect or back titration and if the rate of complex formation is slow or there is any difficulty in keeping the metal in solution, the latter procedure is to be preferred. Also, if there is no indicator available for the metal or the end-point is unsatisfactory, an indirect titration may overcome the difficulty.

In Experiment 3 practical details are given for the determination of nickel by addition of excess standard E.D.T.A. and back titrating with a standard copper solution at pH 5 using PAN indicator. A direct titration is not possible in this case as the nickel—E.D.T.A. complex is formed slowly.

Experiment 3

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Standard copper solution—approximately 0·01 M. The copper solution may be standardized by a direct titration with the E.D.T.A. using PAN indicator.

PAN solution—0·1 g. of the solid indicator dissolved in 100 ml. ethanol.

Buffer solution pH 5·3 g. sodium acetate dissolved in water with 6 ml. N HCl and made up to 100 ml.

Procedure

At least twice the volume of E.D.T.A. required for complete complexing is added to an aliquot of the nickel solution and, if necessary, the pH is adjusted to approximately 5 with dilute nitric acid or ammonia using a little universal indicator to show the correct pH. Five ml. of the buffer solution is then added followed by a few drops of the PAN indicator and the solution at this stage will be yellow. The excess E.D.T.A. is back titrated with the copper with a colour change to violet at the end-point.

Various precautions may be taken to ensure complete formation of the metal—E.D.T.A. complex. If difficulty is expected, it is best to warm the mixed solutions for a few minutes before adjusting the pH and it is wise always to add a large excess of E.D.T.A. to favour complex formation.

Many of the indicators and their metal complexes are insoluble in water and the titration has to be carried out in alcohol or alcohol-water mixtures. Normally methanol, ethanol or mixtures may be used. As an example, details are given in Experiment 4 for the determination of aluminium. Basically it is a back titration similar to Experiment 3 but dithizone indicator is used and the final titration is carried out in a 50 per cent alcohol-water mixture.

Experiment 4

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Standard zinc sulphate solution—approximately 0·01 M. The zinc solution may be standardized by a direct titration with E.D.T.A. as described in Experiment 1.

Dithizone solution—0.025 g. of the solid indicator dissolved in 100 ml. ethanol.

Buffer solution pH 4·5—5·7 g. glacial acetic acid and 7·7 g. ammonium acetate dissolved and made up to 100 ml. with water.

Procedure

At least twice the volume of E.D.T.A. required for complete complexing is added to an aliquot of the aluminium and the mixed solutions heated to about 90° C. for a few minutes. After cooling the pH is adjusted to 4 to 5 by the addition of dilute nitric acid or ammonia using a minimum quantity of universal indicator to show the correct pH. Ten ml. of the buffer solution is then added, followed by sufficient alcohol to make the final solution a 50 per cent water-alcohol mixture. About 2 ml. of the indicator is added for every 100 ml. total volume and the solution has a greenish-yellow colour. The excess E.D.T.A. is titrated with the standard zinc until the colour changes to red. If conditions are correct, the colour change is very sharp.

One advantage of the back titration technique is that the accuracy may be improved by obtaining a mean value from several end-points. The first end-point is

obtained, a little standard E.D.T.A. is added from a second burette, then metal solution to a new end-point and so on.

Another method of overcoming the disadvantages of the direct titration is by using a replacement reaction. In this, instead of E.D.T.A. itself being added to the metal solution, a relatively weak metal—E.D.T.A. complex is added, the metal being chosen as one which gives a good end-point. For example, an excess of magnesium—E.D.T.A. may be added to a metal solution.

$$M^{n+} + MgY^{2-} \rightarrow MY^{(n-4)+} + Mg^{2+}$$

The metal displaces the magnesium from its relatively weak complex and the equivalent magnesium is then titrated. This is extremely useful in many cases where complex formation is slow. The method can also be used in cases where complex formation is fast enough but a good end-point cannot be obtained. For this, it is not necessary to add excess magnesium but merely sufficient to ensure a good end-point, a technique known as partial replacement titrations. Details are given in Experiment 5 for the titration of barium with a partial replacement technique using magnesium. The barium which does not give a good end-point is complexed first followed by the magnesium which gives a good end-point.

Experiment 5

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.
Standard magnesium solution—approximately 0·01 M.
Buffer solution pH 10—as in Experiment 1.
Eriochrome Black T solution—as in Experiment 1.

Procedure

Five ml. of the standard magnesium solution is added to an aliquot of the barium solution and the pH adjusted to approximately 10. Five ml. of the buffer solution and a few drops of indicator are then added. The solution is titrated with the standard E.D.T.A. until the colour changes from violet to blue. The end-point may be improved if the solution is warmed to about 60°C. but too high temperatures or too prolonged heating will cause breakdown of the indicator.

The E.D.T.A. added is equivalent to the sum of barium plus magnesium of which the magnesium is known. For total replacement reactions it is necessary to use a standard magnesium—E.D.T.A. solution. This can be prepared conveniently by titrating a 0·1 M. E.D.T.A. solution with magnesium. For total replacement titrations, the exact strength need not be known as long as an excess is added.

The experiments quoted have been selected as being typical and it is possible to analyse almost all of the metals in the periodic table by one or other of these methods. Some precautions have to be taken for all of the experiments. If other complexing anions which would compete with the E.D.T.A. are present, they must be destroyed or removed. For example, phosphates will interfere but can be removed usually by anion exchange. In any E.D.T.A. titration it is essential to have the right order of stabilities of the various complexes. Thus the metal—indicator complex must be stronger than any hydroxy complexes but must be weaker than the metal—E.D.T.A. complex. Some metals form exceptionally strong complexes with some indicators and trace amounts of the metal in such cases can ruin an end-point. The difficulty can be overcome by adding a few drops of an even stronger complexing agent to mask the metal. For example triethanolamine will mask aluminium in this way.

End-points are usually better if the total ionic concentration in the solution is low and very often a poor end-point can be improved by merely adding water. For this reason, excess buffer solution should be avoided. Pale colours are preferable to deep ones and very little indicator is required. Aqueous solutions of these indicators are usually unstable and will last for a few days only. It is best to prepare fresh solutions or to add the solid indicator. If the solid is used, it can be diluted with sodium chloride to make addition of the correct amount easier.

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Blaedel, W. J., and Knight, H. T. (1954). Analyt. Chem., **26**, 741.
Taube, H. (1952). Chem. Rev., **50**, 69.

The Polarographic Society

The Polarographic Society now has a permanent address for mail; enquiries concerning the Society should be addressed to:

The Polarographic Society,
c/o Lloyds Bank Limited,
36 High Street,
Wealdstone,
Middlesex.

A meeting is to be held at Wolverhampton Technical College on Wednesday, September 21, 1960 at 2.30 p.m., when the subject will be 'The Application of Polarography in Metallurgical Analysis'. Further details concerning the meeting may be obtained from R. C. Rooney, Esq., British Cast Iron Research Association, Bordesley Hall, Alvechurch, Birmingham.

Appointment of Officers

At the Annual General Meeting of the Association of Public Analysts held at Chester on May 14, 1960, the following were elected as Officers of the Association for the ensuing year:

President	Dr. J. H. Hamence
Vice-President	Mr. D. D. Moir
Past Presidents	Mr. F. C. Bullock
(serving on the Council)	Mr. H. E. Monk
	Mr. J. G. Sherratt
Honorary Treasurer	Mr. R. C. Spalding
Honorary Editor	Dr. E. C. Wood
Honorary Secretary	Mr. F. A. Lyne
Asst. Honorary Secretary	Mr. D. F. H. Button.

APPARATUS & DEVICES

A MODIFIED TANK FOR PAPER ELECTROPHORESIS

by D. A. Osborn

Department of Pathology, Institute of Laryngology and Otology, University of London

THE soaking of the strip in buffer prior to application of material for analysis has always been a major problem which has not been lessened with the introduction of cellulose acetate membrane. Many workers, perhaps the majority, soak their strips in buffer prior to insertion in the tank, removing the excess by gentle blotting. With such a technique the achievement of an even distribution of buffer, so essential to the production of good ionograms, is obviously dependent on the combination of skill and chance and evaporation is difficult to control. Loss of water from this cause leads to a concentration gradient of electrolyte across the strip, being highest at the edges with consequent reduction in the volts per centimetre in this region. The result is a progressive increase in curvature of the migrating band of material. Some workers prefer to place their strips dry in the apparatus and allow soaking to take place by capillarity until equilibrium is reached. In all the conventional tanks the atmosphere is never saturated with water vapour at the commencement of soaking and hence water loss due to evaporation still occurs even before electromigration. With cellulose acetate membrane the effect of such conditions is particularly marked and Kohn (1957) minimized this by covering the under surface of the lid of his tank with 'Wettex' which was kept permanently moist.

The apparatus described here provides for the soaking of the strips *in situ* in a pre-saturated atmosphere. It also incorporates a simple method of maintaining tension on the strips, and retains relative cheapness and simplicity of construction within the scope of the average worker. The dry strips, either filter paper or cellulose acetate membrane, can be easily inserted and removed. The rigid glass top forms a stable platform for the jig used in the micrometer method of application described by Osborn and Bawden (1959).

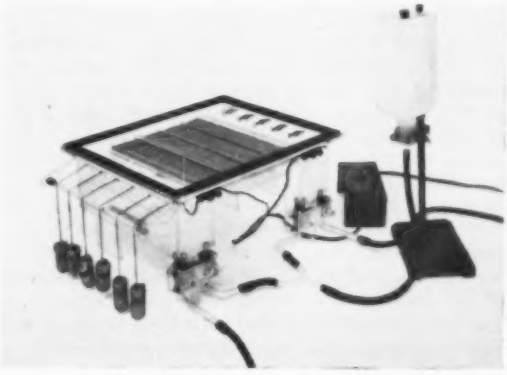

Fig. 1.

Constructed in Perspex (Fig. 1), the tank is designed to take six strips 2·5 by approximately 20 cm., placed horizontally. The platinum electrodes (approximately $3 \times \frac{1}{2} \times 4/1000$ in.) are housed in separate compartments (A in Fig. 2), connected with the strip compartments (B) by removable buffer/agar bridges (C), and the central portion of the tank is raised to reduce the air space. All four buffer compartments are provided with

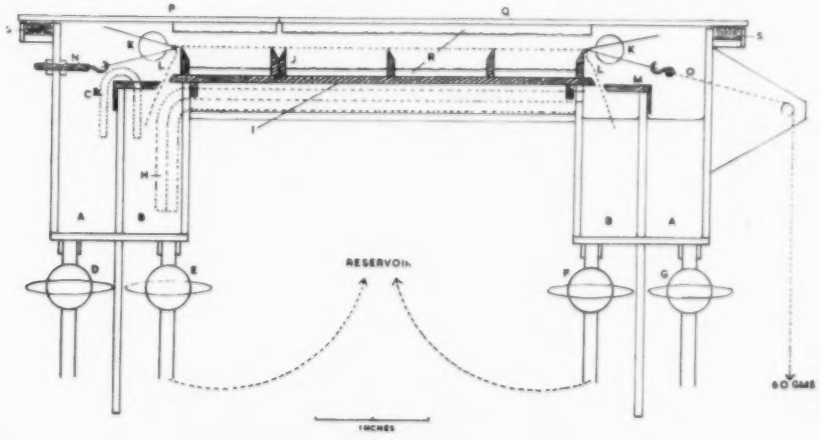

Fig. 2.

drainage holes controlled by stopcocks (D,E,F and G). These serve not only for emptying and cleaning, but in addition, those from the strip compartments are connected through a Y-piece to a reservoir thus enabling the level in these compartments to be raised or lowered at will. Internally, the same two compartments are connected by a capillary siphon tube (H) above which a removable platform (I) carrying 'knife-edges' supports the strips. A double 'knife-edge' (J) supports the strip in the region where material is applied, to ensure that the strip is completely flat at this point. Contact between strip and buffer is by means of a separate filter paper wick of the same width secured to the ends of the strip by one inch 'bulldog' clips (K). The wicks (L) pass downwards through a narrow gap in the detachable roof (M) of the buffer compartment (B), and the clips are secured at one end to an adjustable hook (N) in the end wall of the tank and at the other by means of a thread (O) passing through a fine hole in the wall to an external weight of 60 g. The lid consists of approximately '20 oz.' sheet glass in two sections (P and Q) with unbevelled edges. When placed together on top of the tank the junction lies over the region where material will be applied to the strips and is normally sealed by Sellotape. The under surface of both sections is covered by 'Wettex' cloth (R) and similar material lies between the 'knife-edges' of the removable platform*. The cloth is kept permanently moist. The lid is clamped against an external rim of sponge rubber (S) by means of large 'bulldog' clips.

Operation of the tank is simple. The level of buffer in the strip compartments (B) is lowered by means of the reservoir, then the dry strips previously assembled with their wicks and clips are placed in the tank under tension but with the wicks not in contact with the buffer compartment contents. The lid is placed in position and clamped down. After one and a half to two hours, during which the atmosphere in the tank becomes saturated with water vapour, the level of buffer is raised again and the taps E and F closed. The greater part of the wicks is now covered and the strips become evenly soaked by buffer from either end, the time taken for equilibrium to be established depending on the nature of the strips. With 3MM paper three hours is sufficient but with cellulose acetate membrane 15 hours or overnight soaking is necessary. Because of the shorter period of electromigration with cellulose acetate membrane, the total time is about the same for both types of strips. The strips are now ready for application which is carried out by removing the Sellotape from the lid and sliding the smaller section a few millimetres to give access to the strips. During the operation the greater part of the gap is covered by wet filter paper. As soon as application is complete the section is slid back into place again and the Sellotape replaced. After electromigration the strips are removed from the tank by their clips and when these are released the wet wicks automatically fall away leaving the strip ready for fixation and subsequent processing.

* More recent experience has shown that Wettex below the strip is not essential, but if used it should be kept well clear of the knife edges which should be smeared with silicone grease to prevent an electrical leak.

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PLASTIC FILMS AND MOULD GROWTH

by L. D. Galloway

IT is well known that the various types of plastic films now available differ widely in their transmission of moisture vapour and of gases such as oxygen and carbon dioxide.

In selecting packaging materials these differences must be borne in mind. Too high a moisture transmission may cause loss in weight of the packaged product. On the other hand, a *loose* wrapping of moisture-proof material (such as those used for certain 'rindless' cheese) leads to surface condensation and a high humidity which encourages mould growth.

One might expect that a *closely* fitting moisture-proof wrapping would check both moisture loss and

mould growth; the accompanying photograph indicates that this is not necessarily so. To prevent growth of mould on the surface, even of a material in close and continuous contact with the film, it is necessary to use a film that is reasonably air-proof.

The two small bags illustrated are of polythene and cellulose/polythene respectively. They are partly filled with a nutrient jelly containing 2 per cent agar and inoculated with mould spores. In the head space of the bags growth and sporing of the mould occur normally. No growth has occurred on that part of the gel in *contact* with the laminated film. But with the polythene film—allowing some oxygen transmission—a white skin of fungal mycelium covers this surface; sporing heads do not develop, either because the air supply is limited or because their formation is prevented mechanically.

This ability of mould fungi to grow at a film/nutrient interface does not appear to have attracted attention, and therefore seems worthy of photographic record.

A NEW pH METER CELL ASSEMBLY

by P. Bamford

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IT is often necessary in laboratory routines to adjust the pH of a large number of small aqueous samples to a standard value. This operation is somewhat tedious with conventional pH meter cells such as the Morton cell. This note describes an inexpensive apparatus which simplifies and speeds up this kind of procedure.

The cell is a glass cylinder fitted with a tap at the bottom, and a filling funnel at the side. A short rubber bung carries a glass electrode, a calomel electrode in a KCl reservoir with sintered glass plug, a thermometer and four glass capillaries conveying the necessary reagents (Figs. 1 and 2).

This assembly is mounted on a vertical slotted bar pivoted at its upper end. In the slot slides a small ball race which is attached eccentrically to a wheel driven by a low voltage electric motor. This runs from a battery charger normally used to recharge batteries for a 12 volt Cambridge bench type meter. (Any small electric motor can, however, be used, as no interference has

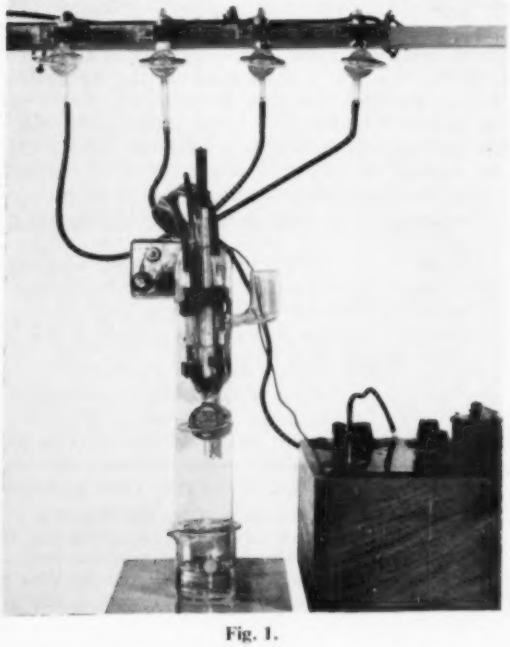

Fig. 1.

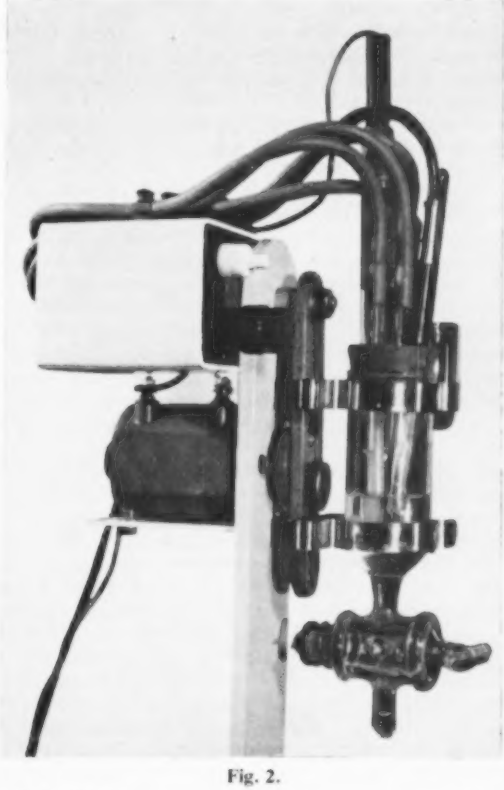

Fig. 2.

been experienced even with mains equipment). The motor is controlled by a push-on, push-off switch, and a small resistance in series brings the speed down to *c.* 130 oscillations per minute. The cell can be used for samples of 6 to 20 ml. The rubber tubes to the capillaries are constricted by a screw-clamp so that reagent is added from the stock bottles drop-wise when the controlling stopcocks are opened.

In use, the pH meter is standardized in the normal way, the test solution then poured in and its pH read; with the shaker running, the appropriate reagent is run in until the desired pH is reached. The shaker is stopped and after the test solution is run off, the cell is washed in preparation for the next sample.

ACKNOWLEDGMENT.—*The help of Mr. S. Barton in the construction of the equipment is gratefully acknowledged.*

A SIMPLE TEST FOR EFFICIENCY OF PACKS FOR FOODSTUFFS

by W. G. Wearmouth

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IN recent years there have been considerable developments in the packaging of foodstuffs in flexible packaging materials such as polythene, laminates of cellulose with various plastic films, and so on. In many cases, as for example in the packaging of dairy products, the film used must have an extremely low oxygen permeability so that spoilage of the foodstuff due to oxidation effects can be reduced to a minimum. Such permeability characteristics of the films can be measured with a fair degree of accuracy to enable a sound choice of film to be made.

In addition, however, once the film has been selected in this manner it must be possible to seal the film completely around the portion of the foodstuff to be packed. If this is not done successfully, air will leak through the gaps in the seals (whether these be metal seals or the more conventional heat seals) and the finished pack will be useless. In some cases (the so-called vacuum packs) it is customary to remove much of the residual air in the pack before the final sealing is carried out. The resultant pack, being under a partial vacuum, rapidly allows air to pass through any leaks or pin holes in the seals of the pack.

A simple non-destructive test has been devised by which such faults in the sealing and also faults (such as pin holes) in the film can be rapidly spotted in the freshly prepared pack. The finished pack is immersed in distilled water contained in the lower half of a vacuum desiccator; a weight is attached to the pack by a piece of string to prevent the pack from floating, the lid of the desiccator replaced and the apparatus is then attached to a high vacuum pump. After the vacuum has been applied for a minute or so a stream of air bubbles will be seen coming from any weak spots in the seals or any accidental flaws in the actual film used for making the pack.

This test is most effective for locating faults in sealing techniques. It is also valuable for proving the efficiency of the packing before test packs are put aside for long storage trials. A rough diagram, not to scale, in Fig. 1 illustrates the type of apparatus used.

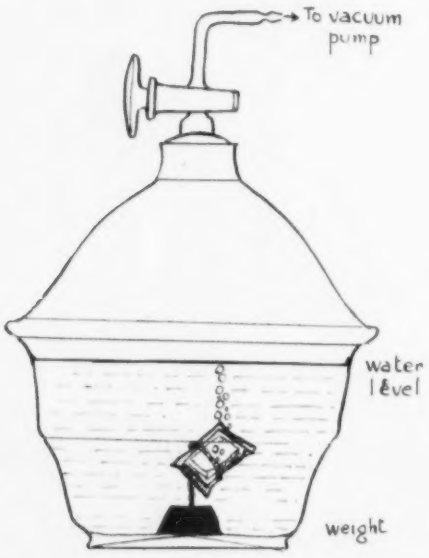


Fig. 1.

APPARATUS AND DEVICES

It is evident from letters received that the Apparatus and Devices Section of *Laboratory Practice* arouses considerable interest. We should like to point out that we are at all times pleased to receive from readers notes, however short or simple, for inclusion in this section.

BOOK REVIEWS

Zone Refining and Allied Techniques, by N. L. Parr, M.I.Mech.E., F.I.M. London: George Newnes Ltd., 1960. 40s. pp. 184 + xvi.

This book sets out to review the technique of zone refining and to outline practical methods associated with this and allied processes. The second function is performed with credit.

One senses that the author felt he must provide some theoretical background and this he stretches into the first 17 pages, by virtue of much repetition of the principles of equilibrium in metallic alloys and the mechanisms of solidification. The practical aspects occupy Chapters 3 to 8, 90 per cent of the book, and are very well presented. The illustrations are detailed, the plates beautifully reproduced, and the wealth of information makes this book invaluable for the practical man. There is still a tendency for repetition, even in these later chapters, and the author leaves little to the imagination by including everything. For example, Fig. 20 shows some cross sections of containment vessels, and includes a circle, a square, a rectangle, a U-shape, a V-shape, an annulus, and combinations of these. I would have expected any scientific assistant to be capable of working this out for himself.

Occasionally the author has extended the title of his book, as in Chapters 7 and 8, on the examination, properties and application of high purity materials. By definition, a book of this size can only present a very sketchy outline of these aspects, and hence the references are few and not sufficiently representative. Fig. 112, for example, a vacuum extensometer, is a complex method of achieving an objective and only one among many possibilities.

However, the presentation of zone refining and single crystal techniques is admirable, and I consider the price quite reasonable for those who may be involved in this field of work.—C. R. TOTTLE.

Principles and Methods of Clinical Chemistry for Medical Technologists, by Eugene W. Rice, Ph.D. U.S.A.: Charles C. Thomas, Illinois. Oxford: Blackwell Scientific Publications Ltd. 1960. 56s. Pp. 286 + xvii.

The aim of this book as stated in the preface is 'to teach the student to apply his training in basic chemistry to clinical chemistry in order to perform the analyses intelligently rather than mechanically'. The book consists of two sections, one on the principles underlying the usual laboratory operations of clinical biochemistry, and a practical section giving a good range of routine methods. The whole book bears the marks of being a transcription of lecture notes, and this may account for some of its failings as well as its virtues: a set of very good lectures which have lost some of their clarity and directness in the transcribing. Thus the useful but disproportionately long chapter on complexometric titrations may have originated in a stimulating lecture explaining this recent development to students who would not yet find it in their text-books of analytical chemistry. Milliequivalents which would normally be introduced early, together with equivalent weights, are relegated to the section on electrolyte balance on p. 102; this seemingly illogical procedure becomes intelligible when seen as a subject introduced during a course of lectures precisely at the stage when students first need this knowledge and are ready to digest it. Even so, this approach is open to criticism: milliequivalents could be accepted as the simple and natural units they are and be explained as such from the start, rather than beginning with the arbitrary notation of milligrames per 100 ml., converting these into milliequivalents and reducing them in the student's mind to the complication of a complication. Again, degrees of gastric acidity, defined here as ml. of 0·1-N sodium hydroxide required to neutralize 100 ml. of gastric juice (p. 114), are simply milliequivalents of acid per litre.

There are a few signs of hasty assembly into book form, as evidenced by the table of atomic weights on the inside front cover where a footnote explains the meaning of values in brackets but no such values appear in the table, or by the bromsulphthalein test on p. 149 where the ward procedure is given only under 'Interpretations'. A number of minor errors or indefinite wordings have also entered the book, quite admissible in *extempore* lecture notes but less so in print, e.g. the continued distinction between c.c. and ml. (p. 10), the term 'solution of colloidal gold' (p. 121), the statement that Bence-Jones protein (defined here by its thermal solubility properties) is excreted in most cases of multiple myeloma (p. 126) or that serum salicylate levels of over 15-20 mg./100 ml. are toxic.

These are points of detail, suitable for the author's errata list, in a book that is well planned, with the clear aim of enlarging the horizon of student medical technologists. The book will be of greatest use for teachers planning a set of lectures and course work who could use it as a basis to expand and clarify. It can also be read with profit by students when revising course work already done. The experimental section commands attention on account of its up-to-date selection of methods.—A. L. TARNOKY.

Dictionary of Cinematography and Sound Recording, by Wallace S. Sharpe, M.B.K.S., F.Inst.D. London: Fountain Press, 1960. 15s. 6d. pp. 144.

The book is in two parts: Part 1 (63 pages) consists of data on a variety of topics—for example, sound; light and lighting equipment; film projection. Part 2 (65 pages) is a dictionary of terms used in amateur and professional cinematography and recording.

The stated aim is to provide a means of accurately defining and explaining standard terms, and, in the data section, to provide valuable technical information for amateurs and professionals.

The rapid technical development in film-making and sound recording in recent years, together with their

increasing use in science and industry, make it important that an accurate and effective source of reference and definitions should be available. It is unfortunate, therefore, that this book is only partly successful. Although there is much useful information in the data section, those chapters on colour and sound, in particular, are inadequate. There are some surprising errors. For example, the lens projection charts for 8 mm. and 9·5 mm. film on page 67 lack the vital information concerning the focal length of the projection lenses required. The dictionary, too, has a number of useful definitions but there are surprising omissions and inaccuracies. The definitions cover a range of advanced technical subjects (a magnetostriction loudspeaker is defined as one using a material having magnetostrictive properties—but there is no definition of magnetostriction itself). This section also includes studio terms and jargon ('N.G.' is defined as the abbreviation for 'no good'). Apart from occasional obscurity in definition, the least satisfactory aspects of this section are the inaccuracies—a bulk eraser is defined as 'a device designed to pass a high intensity alternating current through wound reels of magnetic tape'. There are a number of inexplicable omissions; there are no references to colour film processes or principles and no mention or explanation of wide screen systems.

Although there is much useful information in this book, it is neither as completely comprehensive nor as accurate as might be desired. Its principle value will be to the amateur cine enthusiast rather than to the technician or professional film maker.—BRIAN W. COE.

Encyclopaedia of Microscopic Stains, by Edward Gurr, F.R.I.C., F.L.S., M.I.Biol., F.R.M.S. London: Leonard Hill (Books) Ltd. 1960. 95s. pp. 498 + xii.

If Mr. Gurr had aimed to write a dictionary rather than an encyclopaedia this book would have served its purpose equally well, with considerable economy in respect of size, and presumably of cost. It is where the work is most encyclopaedic that it is least valuable.

Basically it consists of a list of dyes, arranged in alphabetical order, with synonyms (admirably indexed), structural formulae and solubilities all clearly set out. So far so good, and indeed, very good; but at this point Mr. Gurr proceeds to justify the title of encyclopaedia by telling us all he thinks worth knowing about each dye, and the result is a sad mixture of commercial information, no doubt accurate, but distinctly out of place, combined with a selection of references from scientific literature, supported by an 18-page bibliography, most of which are valueless, and some, frankly misleading. The cumulative effect of these passages is one of irritation, growing to exasperation and tempered only occasionally by amusement. The latter emotion is sometimes aroused by encounters with both the Iron Curtain and the Bamboo Curtain in stain technology: Ethyl purple 6B 'is only in occasional demand, and then usually only in very small quantities in certain areas of South-Eastern Europe', but it is gratifying to know that it 'could no doubt be substituted for Spiller's purple or for dahlia' even though, regrettably, it 'does not appear to offer any particular advantage over either'. On the other hand, it is South-Eastern Asia that uses Benzyl Green 'for both animal and plant tissues,' but only occasionally, and 'there are no reports available regarding its use.' *No* reports? We have a feeling that Mr. Gurr must be holding out on us somewhere, especially as he drops the hint that: 'It might be of interest as a contrast to various nuclear stains.' Imagination boggles at the purposes to which a Siamese might put a bright green water-soluble dye.

All this is entirely without any hint of literary or other source, whereas, in total contrast, we have Fast Acid Blue, which 'was employed by Romell, L. G. (1934) in his technique for soil bacteria.' A few of the lines squandered on market research in the previous quotations might have been used to tell us what Romell actually did.

Bismark Brown Y is employed '*as a component*' (my italics) of Neisser's stain, and the only reference to the HCl –Giemsa technique, at the end of two and a half pages on Giemsa, is that Knaysi (1958) gives us to understand that it is no use. This will surprise many people who employ the method. However, when we examine the bottle, we find that R. 66 Giesma is marketed, not by the author of the book, but by another person of the same surname. Possibly Dr. Knaysi used the wrong sort of Giemsa.

We learn that confusion has arisen between haematoxylin and hematoxylin, sometimes referred to as haematoxylin; it certainly is a little confusing.

As a book of rapid reference on the subject of the dyes themselves, this work is most admirable, and should find a place in every scientific library; but those seeking its guidance should not rely too much upon the author's interpretations of the scientific purposes to which the various stains have been put.—K. A. BISSET.

Making and Printing Colour Negatives, by John Vickers, F.I.B.P. London: Fountain Press. 1960. 30s. pp. 194.

There is no doubt that for better or worse, we are now launched on the pursuit of the perfect colour print. Whatever that may mean, and the more we study the fascinating subject of human vision, the more we are made to realize how individuals differ in their evaluation of colour terms. However, so great is the differential value of colour in scientific and applied photography that it is only to be expected that the demand for its use in printing will continue to increase.

Therefore an authoritative publication such as Mr. John Vickers's book on the production of colour negatives and colour printing fulfils an urgent need.

The author deals with his subject in an extremely practical manner and in terms such that both amateur and experienced workers will find it a most useful guide.

Divided into five parts and 25 chapters the work opens with a discussion on vision and illusion, the negative colour process and the characteristic curve.

Mr. Vickers places great emphasis on the fact that colour fidelity is largely an illusion in all known methods of colour photography, and that this is inevitably bound up with the mechanism of human vision itself. He goes on to state that our aim is to produce the illusion of fidelity, and proceeds to explain how this aim may be achieved. The function of corrective masking etc., is dealt with fully and with clarity in this section.

However, the author's statement with regard to the inferiority of copies of reversal transparencies is no longer valid, since at least one of our leading manufacturers has introduced a copying service which very often produces results even superior to the original, especially in the shadow areas, due to the incorporation of an electronic masking device in their copying technique. This is just one of those things in this swift moving age of scientific progress.

Part II forms a most useful guide on exposure of the negative by daylight, tungsten illumination, flash bulbs, and electronic flash.

Sufficient data is given on the use of filters, contrast ratios, reflectors and fill in flash in daylight to be of real value without being tedious.

The information on types of tungsten lamps, lighting equipment, the use of light meters for determining exposure, etc. includes notes on an extremely simple CT comparator, for which the author claims that a difference of 100°K. can be detected, which is quite adequate for modern colour negative materials.

There follow chapters on techniques with expendable flash, electronic flash, and mixed lighting which should enable anyone conversant with black and white work to make a start in this field of colour work, although the technique of electronic flash could have been expanded with advantage.

The latter part of the book is devoted to negative processing and colour printing, and covers Kodak Ektacolor and Kodacolor with useful hints on solution storage although there is a slight repeat of information on page 96.

There are also notes on the processing of Icicolor, now obsolete, and Agfacolor, Raycolor and Pakolor in which the author points out the wisdom of using the manufacturers' formulae as opposed to the use of substitutes at this stage in colour work.

Sources of trouble in the darkroom are also dealt with, together with suggestions for equipment and a warning against dermatitis, to which some people are susceptible, together with precautions for its prevention.

In colour printing the false economy of using doubtful or stale materials is rightly stressed. Unless the reader places a very low value on his time it is much wiser to buy and use colour materials of which the age and storage conditions are known.

Practical advice is given on the testing of washing efficiency, darkroom safe lighting, and projection printers, etc.

Colour printing procedures both additive and subtractive are covered at great length and in such a manner that anyone of average intelligence cannot fail to grasp the essentials of colour filtering.

A chapter is devoted to the processing of Agfacolor and Ektacolor papers, where it is pointed out that other manufacturers' papers are treated in sequences which are very similar to the Agfacolor process.

Directions are also given for drying, glazing and mounting.

The rest of the work deals with masking techniques, retouching, the copying of colour transparencies and making of separation negatives.

The work is illustrated throughout with diagrams and colour plates and forms a most valuable addition to the library shelf of both the beginner and experienced worker.—NORMAN GRUBER.

Manuale lactis, by M. E. Schulz and G. Sydow. *Cheese* (Folge II, Klasse 5, 1955); *Milk products—By-products* (Folge II, Klasse 6, 1957); *Dairy equipment* (Folge II, Klasse 7, 1959); Hans Carl, Nuremberg.

The first series of this comprehensive abstract compilation began in 1948, and these latest three volumes are in the second series which began in 1952. The object of the authors has been to give a brief abstract or at least the title of every worth-while paper published since the issue of the first series. The abstracts are classified according to the well-known *Bibliotheca lactis* of the senior author. This is very simple and a key is provided in German, English, French, Spanish, Danish, and Dutch at the beginning of each volume. The abstracts are then listed in each section alphabetically under the names of the authors. The publications may therefore be regarded as a compressed form of *Dairy Science Abstracts* covering several years and arranged according to an easily used system. All scientists and technologists today should be able to read German, and so these volumes are invaluable to all scientists interested in the dairy industry. Each section is in effect a brief review of one aspect of dairy science.

The books contain no indexes, either of authors or of subjects. Doubtless the authors considered that these were unnecessary. Nevertheless, an author index would not be without its usefulness and this might be considered for future issues.—J. G. DAVIS.

Atomic Energy Appointment

The U.K. Atomic Energy Authority announce the appointment of Mr. B. S. Smith as Head of the Isotope Research Division of A.E.R.E., Harwell. This Division is situated mainly at the Wantage Research Laboratory at Wantage, Berkshire, a few miles from the Harwell site.

Mr. Smith, whose appointment dates from May 17, 1960, was formerly Head of Health Physics Division at the Atomic Energy Research Establishment, Harwell, and his successor in that post is Mr. N. G. Stewart, at present at the Authority's Dounreay Experimental Reactor Establishment. Mr. Stewart will take up his new duties on August 1, 1960.

Laboratory Note-book

Donations wanted for Prize Fund

Since its formation in 1948 as the Science Technologists' Association, the Institute of Science Technology has steadily increased its membership and it is now firmly established as the professional organization to which it is appropriate for the science laboratory technician to belong. The Institute is solely concerned with examination and certification and seeks by these means to provide a high standard of training and professional standing which will encourage young people to take up laboratory work as a career. The acute shortage of skilled technicians is well known to all who work in science and technology and the problem can only be resolved by systematic courses of training leading to accepted qualifications.

Some measure of the Institute's significant contribution to the training of the technician may be shown by the implementation of a new scheme of qualifying examinations in 1960 in conjunction with the City and Guilds of London Institute and, in this connection, the Council of the Institute have agreed to the formation of a Trust Fund, the income from which will be devoted to the award of prizes. In this way, it is hoped to give further encouragement to boys and girls entering laboratory work. Four members of Council have been appointed to act as Trustees of the Fund and the investments will be held by the Official Trustee of Charitable Funds. All income will be free of tax.

When sufficient donations have been received, the Deed of Trust will be signed as follows:

'Upon Trust for the advancement of Education in Science and Technology by the award of Prizes (including Medals) in accordance with the rules to be made by the Council of the Institute of Science Technology to persons who, in the opinion of the Trustees, have passed public examinations with credit, or who have written an article published in the Journal of the Institute or, if the said rules so provide, in any other publication which appears to the Trustees to be of sufficient merit.'

Provision has already been made to award the following prizes:

1. Two awards of Six Guineas for the best performance in the Certificate and Advanced Certificate Examinations held annually by the City and Guilds of London Institute in conjunction with the Institute of Science Technology in the Laboratory Technicians' Course.

2. An annual award of a Medallion together with a book token to the value of Two Guineas for the best article published in the Journal of the Institute.

For this purpose the sum of £500 will be required and those interested in supporting the Fund are invited to make a donation.

All donations should be sent to R. S. Diaper, Hon. Treasurer, Institute of Science Technology, 19 Hammond Avenue, Mitcham, Surrey.

Do-it-yourself Anatomy

An anatomical model of the human body, sculptured in plastics, and standing 16 in. high and is approximately one-fifth life size is marketed by Griffin & George Ltd.

The model has three distinct divisions, the skin, the skeletal system and the internal organs which can be taken apart in that order, thus providing an instructional introduction to human physiology for the student and interested layman alike.

The skin is represented by a layer of pink tinted plastics constructed in three removable parts which clip together and are: the rear and front halves, and the breastplate. Embossed on to the inside of the skin is the circulatory system of the head, trunk and limbs, and the musculature of the trunk and the limbs.

The skeletal system is built to represent bone in white plastics with the main joints articulated. The crown of the cranium is removable to reveal the brain, as is also the front of the rib cage which reveals the entire internal organs.

This transparent model can be dismantled organ by organ.

The internal organs show in detail the respiratory system with the construction of the lungs, and the heart, the front half of which is removable to show the chambers. The digestive system, including the stomach, pancreas, the small and large intestines and the rectum is also shown. The excretory system shows the kidney, which opens to expose the workings, and the urinary bladder. All parts of the internal organs are removable through the breastplate.

The model is supplied as a set for assembly, and requires only a small tube of polystyrene cement for construction. The instructions for assembly come with a ten page booklet entitled 'An Introduction to Human Anatomy', which accompanies each model.

Colour Television

A unique demonstration was presented at The Institution of Electrical Engineers recently when two French authors, R. Chaste and P. Cassagne, described a new system of colour television transmission. Scenes originating in Paris were seen in full colour by a large audience in London, on screens which differed little, if at all, in their external appearance from those of ordinary 'black and white' television receivers.

Moreover, the transmitted pictures were 'compatible': that is to say, if put into a colour receiver, they gave a coloured result; if put into a normal television set, they gave a black and white picture. This 'compatibility' is important, because it would provide a means of bridging the gap if a colour service were to be introduced, so that until a user bought a colour receiver, his existing set would still give him a black and white result from the colour transmission.

Behind this feat lay a triumph of engineering co-operation. Experts in the French companies responsible for the development of the system, the French Posts and Telegraphs, the Radio Television Française, the British Post Office and the B.B.C., together set up a complicated radio link comprising sections of the existing Eurovision network, with additional microwave links of the type used by the B.B.C. As an additional precaution, a video recording was made in London beforehand of the complicated colour signals transmitted from Paris, to be used in the event of their being any breakdown in the radio link between the two capitals. It is satisfactory to report that the recorded version was not in fact required.

LABORATORY EQUIPMENT & APPLIANCES

Machlett OEG-60 beryllium window tube for research and fluorescence analysis.

Machlett Tubes for X-Ray Fluorescence Analysis

New x-ray tubes for x-ray fluorescence analysis in which the characteristic radiation from impurities in the tube anode is reduced to a very low intensity are now being produced by Machlett Laboratories Inc.

The spurious characteristic radiation generated in x-ray tubes is one of the factors limiting the sensitivity of the x-ray fluorescence method. The new tubes allow an improvement in sensitivity to be obtained, especially for copper and nickel, and the effect of target impurities is now insignificant.

The improved x-ray tubes, designated AEG-50-S, OEG-50-S and OEG-60-S, have been developed from the well-known AEG-50, OEG-50 and OEG-60 types, with which they are interchangeable. The AEG-50-S and OEG-50-S tubes have 5 mm. × 5 mm. foci and are available with tungsten or platinum anodes. The OEG-60-S tube has a 6 mm. × 6 mm. focus and is available with tungsten, platinum and molybdenum anodes. All the new tubes have beryllium windows and operate up to 50 kV. (Available from: Watson & Sons (Electro-Medical) Ltd., Industrial Division, North Wembley, Middlesex.)

Hydrogen Generator

This apparatus has been especially designed for use in polarography, where a small but continuous flow of hydrogen is required for de-oxygenating solutions prior to polarographic analysis.

It eliminates the inconvenience of transporting and replenishing cumbersome gas cylinders and the difficulty of controlling the flow of small quantities of gas. It consists of a d.c. power pack and a special low voltage electrolytic cell built into a compact self-contained unit. A rotary switch enables hydrogen to be generated at four different rates up to 3,000 c.c. per hour and in polarography 5-10 ml. of solution can be de-oxygenated in four to five minutes. (Manufacturers: Southern Instruments Ltd., Frimley Road, Camberley, Surrey.)

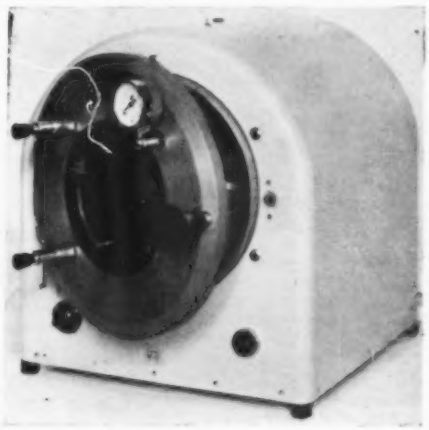
The A. 1653 hydrogen generator.

A range of polytetrafluoroethylene beakers for laboratory or industrial use in the handling of corrosive fluids.

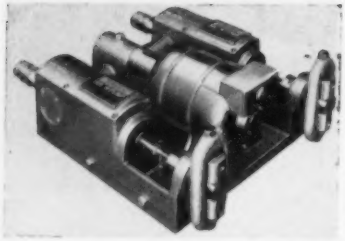
P.T.F.E. Beakers for Corrosive Chemicals

Because of its unique resistance to chemical attack and corrosion, P.T.F.E. has many applications in the chemical processing industry.

One of the latest P.T.F.E. products from the Fluorocarbons Department of A.E.I. Radio and Electronic Components Division is a range of beakers which will find an application in chemical laboratories and industry generally, where corrosive liquids and chemicals are handled. (Manufacturers: Fluorocarbons Department, A.E.I. Radio and Electronic Components Division, 155 Charing Cross Road, London, W.C.2.)



On the right is shown the new vacuum oven being made by D. A. Gunn (Engineering) Ltd.



The Duplex pump accommodates automatic control on both or one of the pump heads.

D.C.L. Pumps

This range of metering pumps has recently been further extended by the development of a Duplex version of the 'M' pump.

Many hundreds of 'M' pumps are in service in the United Kingdom and abroad and by developing from this unit a model which embodies two of the 'M' pump's standard pumping heads driven 180° out of phase from a common motor, the output of the pump is doubled and the flow characteristics improved when the heads are operated in parallel. As an alternative application each head can be operated as an independent metering head pumping two different liquids.

The Duplex pump is designed to accommodate automatic control on both or one of the pump heads. It is available with totally enclosed or flameproof motors and, as with the single head model, the pumps will also accommodate 60-cycle motors by modified gearing.

The maximum capacity of each cylinder is 8·23 g.p.h. at 50 p.s.i.g. with a series of alternative pump heads giving infinitely variable ranges of capacity for smaller volumes and higher pressures.

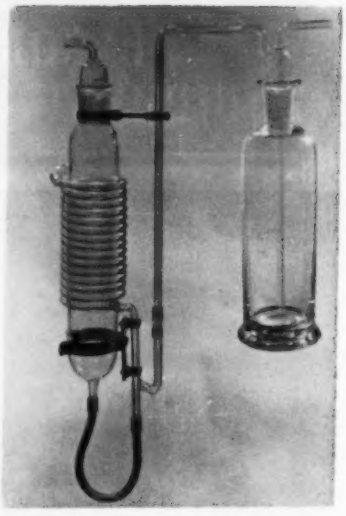
(Manufacturers: The Distillers Co., Ltd., Great Burgh, Epsom, Surrey.)

The Micro-Oven

A handy oven is now available for small scale work and for semi-micro analysis. The micro-oven consists of an anodized aluminium outer casing—to withstand corrosive atmospheres—and an inner metal casing forming a cylindrical cavity 4 in. deep × 4 in. diameter. Fitted to the double walled lid there is a small tray which locates in the centre of the cavity as the lid is placed on the mantle—venting holes are provided in the base and in the lid, with a second hole for a thermometer. High temperature lagging 2 in. thick provides good thermal efficiency and temperatures up to 300°C. can be quickly reached although the loading is 200 watts only. The price is £12.

(Manufacturers: Isopad Ltd., Barnet By-Pass, Boreham Wood, Herts.)

The pocket microscope made by Ellis Optical Co., Mayday Road, Thornton Heath, Surrey.

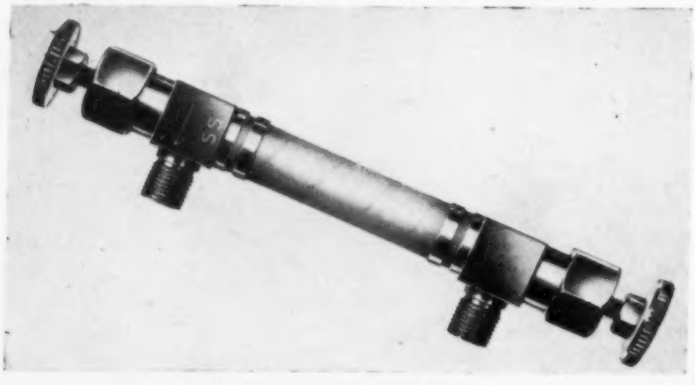


'Nilox' Apparatus

The Nilox gas purification system enables the laboratory worker to obtain on the turn of a tap a continuous stream of hydrogen, nitrogen, argon etc., completely freed of the oxygen normally contaminating these gases as supplied in cylinders. Ordinary commercial nitrogen can be purified to a degree much superior to high purity commercial nitrogen and will reduce an oxygen content of 2,000 p.p.m. down to 0·2 p.p.m.

The gas stream may flow at any rate up to 20 litres per hour and the reagents need only be renewed at long intervals. The two purifiers, which are mounted on a special stand, will also retain impurities such as CO_2 H_2S, SO_2, Cl_2, NH_3 and amines.

(Manufacturers: Southern Instruments Ltd., Frimley Road, Camberley, Surrey.)



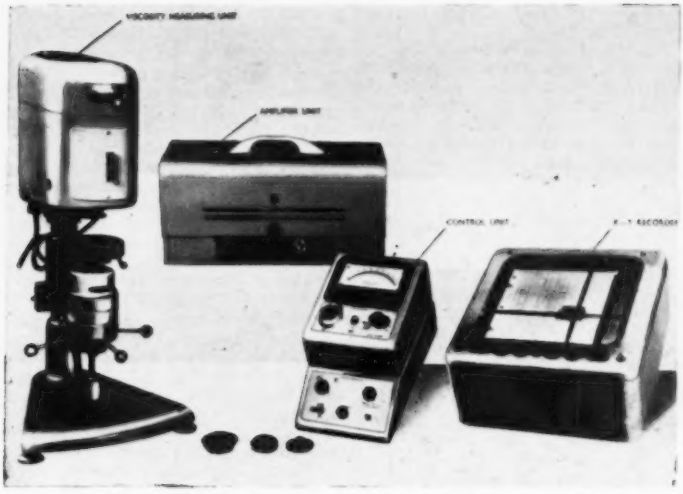
A 2 ml. capacity gas sampling bomb.

Gas Sampling Bomb

A 2 ml. capacity sampling bomb has recently been developed. This bomb has been thoroughly tested by various sections of the oil and chemical industries over the past 12 months, and has now been added to the existing range of Hone apparatus of this kind with a seven to ten day delivery.

These 2 ml. bombs, which are tested to 1,000 p.s.i., are made of a transparent plastic, thereby allowing easy visual check on the liquid level. An anti-static device for complete safety in operation when taking samples of hydrocarbon products has been built into the vessel, which is complete with two fine control stainless steel needle valves.

(Manufacturers: F. J. Hone & Co., Ltd., 19 Eldon Park, London, S.E.25.)



The Ferranti-Shirley viscometer with automatic flow curve recorder.

Automatic Flow Curve Recorder for Ferranti-Shirley Cone-Plate Viscometer

The Ferranti-Shirley cone and plate viscometer, which is widely used in laboratories and industry for studying the anomalous flow behaviour of the more complex fluids, has now been modified to incorporate a new automatic flow curve recorder.

As a result, non-Newtonian flow curves of shear stress against rate of shear can be plotted automatically, thereby eliminating the necessity for constructing graphs by time-consuming manual methods. In addition, it provides a most suitable means of standardizing the test procedure so that operator error due to inconsistent experimental conditions is minimized. This is particularly important for the measurement of time-dependent flow properties, e.g. with thixotropic substances where close control of both the magnitude and duration of shear is essential. The recorder is capable of plotting a complete shear stress/rate characteristic in about 10 seconds for most non-Newtonian fluids.

With the cone and plate viscometer, the fluid to be examined is sheared in a narrow gap between a stationary flat plate and a slightly conical, rotating disc, driven at controlled speeds, ranging from 0·1 to 1,000 r.p.m. by a variable speed motor. The cone speed is directly proportional to the rate of shear. The amount of viscous drag on the cone is shown on an indicator unit and is directly proportional to the shearing stress. Thus, at any rate of shear or shearing stress, the apparent viscosity can be readily determined and flow curves plotted.

The new equipment comprises a flow-curve recorder adaptor unit, designed to fit beneath the existing viscometer indicator unit, so that all controls are readily accessible in one unit, and a Houston Instruments HR-92 X-Y recorder. The latter instrument has been selected for use with the equipment because of its small dimensions, compatible input and performance characteristics and moderate cost. However, most types of X-Y recorder can be used provided they have the correct input characteristics.

In operation, the control unit generates a voltage increasing linearly with time which is applied to the d.c. servomotor in the measuring unit via an amplifier, to accelerate the cone up to a predetermined velocity. The tacho-generator, which is an integral part of the servo-mechanism controlling the cone velocity, supplies the speed signal to the Y-axis of the recorder, calibrated in rev./min. The corresponding torque acting on the cone is measured by a precise dynamometer consisting of a spring and potentiometer which supplies the torque signal to the X-axis, calibrated in dynes-cm. Multiplying by the appropriate cone constants gives the shear stress/shear rate characteristic in absolute units.

On reaching the maximum speed the cone normally reduces speed automatically to zero, but a 'hold switch' has been incorporated to maintain the preset speed constant for any desired time before speed reduction is initiated.

There are eight available rates of acceleration, the cone velocity being swept from zero to maximum speed (10,100 or 1,000 r.p.m.) in a time ranging from 10 to 600 seconds with a linearity of about 1 per cent. Intermediate maximum speeds can be selected by means of a ten-turn potentiometer. However, in applications where the duration of shear must be minimized, e.g. accelerating to a specific cone velocity before commencing the test proper, a rapid acceleration/speed reduction facility with a sweep time of about 1 second is available: this is an overriding switch control, and as such, it may be used to speed the setting up and resetting operations. The sweep voltages are generated by a conventional Miller integrator.

Viscosity or shear stress can be recorded against time at a constant predetermined shear rate, by utilizing the sweep voltage as a time base on the Y-axis of the recorder, with manual adjustment of the cone speed. The decay of stress with time can also be plotted in a similar manner.

Basic price of the flow curve recorder adaptor unit is £184 and the Houston HR-92, X-Y recorder (supplied by Scientific Furnishings Ltd. Poynton, Cheshire) £375.

The standard Ferranti-Shirley cone and plate viscometer is priced at £1,150-£1,200.

(Manufacturers: Ferranti Ltd., Hollinwood, Lancs.)

New Radio Chemical Fume Hood

Duralab Equipment Corporation announces that their new line of fume hoods is now available with constant air-flow, permitting the hoods in the laboratory to function as definite parts of the air conditioning system. In keeping with the streamlined air entrainment exhaust features, the service fixtures are mounted on the sides of the chamber affording a full clear working area.

(Export representatives: Schueler & Co., 75 Cliff Street, New York, N.Y., U.S.A.)

NEWS

General

B.F.M.I.R.A. Elections

At the recent Annual General Meeting of the British Food Manufacturing Industries Research Association, Prof. A. C. Frazer, M.D., D.Sc., F.R.C.P., was re-elected President and Lt. Col. G. R. Harding, D.S.O., M.B.E., Vice President for the ensuing year.

The Lord Tweedsmuir, O.B.E., was also elected a Vice President of the Association.

Vacancies on the Council created by the retirement of Mr. Colin S. Dence, B.A., and Mr. H. C. S. de Whalley, F.R.I.C., M.I.Chem.E., M.I.Biol., were filled by the election of Major J. M. Oke, of J. A. Sharwood and Company Limited and Dr. A. Carruthers of the British Sugar Corporation Limited.

Manufacturers' News

Another Instrument Merger

Dr. P. Dunsheath, C.B.E., D.Sc., LL.D., chairman of the 79-year-old Cambridge Instrument Co., Ltd., has announced an offer by his company for the whole of the ordinary share capital of the 15-year-old Electronic Instruments Ltd. The offer is on a share exchange basis of seven Cambridge 5s. shares for three £1 Electronic Instruments, Ltd. An extraordinary general meeting of Cambridge Instrument Co., Ltd. will be called in the near future to increase the nominal capital from £1,500,000 to £1,650,000, by the creation of 600,000 new ordinary shares of 5s.

This merger between two well-known instrument companies with world-wide interests has been mutually sought and enables the two firms to co-ordinate their complementary research, development, production and market resources, thus creating a more powerful organization to meet the rapidly changing conditions and requirements of the home and overseas markets.

It is intended that Electronic Instruments Ltd. will operate under its own name and that Mr. A. C. W. Norman, O.B.E., will continue as chairman, Mr. P. Goudime, M.A., as managing director, and Mr. D. A. Pitman as sales director, joined by three directors of Cambridge Instrument Co., Ltd. Subject to acceptance of the offer, Mr. Goudime will be invited to join the Board of the Cambridge Instrument Co., Ltd.

Publications

Veale Committee Report

A report on the training of radiological health and safety specialists has now been published ('Training in Radiological Health and Safety: Report of a Committee appointed by the United Kingdom Atomic Energy Authority'—H.M.S.O. price 5s. 6d.). This Committee, under the chairmanship of Sir Douglas Veale (formerly Registrar of the University of Oxford) was set up to consider the national need for specialists to deal with the health and safety aspects of the use of ionizing radiations and radioactive substances. The increasing use of radiations for peaceful purposes has created a shortage of these specialists, and the report makes recommendations about the types of experts needed and their training. It is at present being studied by the U.K. Atomic Energy Authority and the government departments concerned.

The report describes the types of work involved in achieving radiological health and safety, and the kinds of training needed. It then reviews the present position in this country and discusses the responsibilities in the health and safety field of government departments, local and public authorities, hospitals and the medical profession, and nationalized and private industry. An attempt is made to estimate the total number of trained people required during the next ten years.

Methods of training for radiological health and safety staff at three levels are considered. At the top level, university postgraduate courses have already been set up to give a wide training—overlapping the boundaries of medicine, biology, physics and chemistry—to selected graduates in science or medicine. There are courses in the universities of London and Birmingham, and Edinburgh University are planning a course to start next October. The Committee believe that these courses, when fully developed, will be sufficient to meet the need for 20 or 30 top grade specialists a year.

The report recommends that similar courses of a rather less advanced kind should be set up at two or three colleges of advanced technology. At a somewhat lower level again, a scheme is suggested for introducing certificate courses at technical colleges in the autumn of 1961.

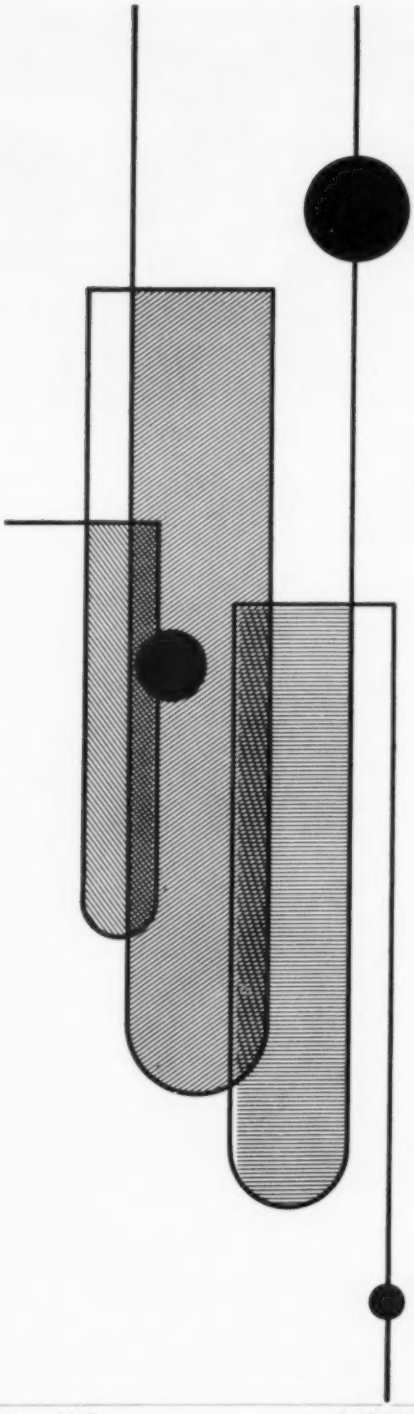
The idea of a national training centre is examined, but the Committee consider it unnecessary, and recommend that the existing educational system should be used to provide the sort of training that is needed.

To make the best use of specialists, the Committee recommend that a national radiological advisory service, with regional centres, should be set up under the Minister for Science. This service would be able to give advice to organizations (such as firms making use of radioisotopes) without full-time health and safety specialists of their own. It would be available to give expert help in the case of an accident involving possible radioactivity hazards. It would also provide such services as processing the film badges which are worn by all workers who may be exposed to radiation.

The report also recommends that all medical students should be given some knowledge of radiological health and safety in their undergraduate training, and that a course should be included in the curriculum for the Diploma in Public Health.

Short residential courses for managers, administrators, medical officers of health and other professional workers are also proposed, as well as books, films, articles, and radio and T.V. programmes for the information of the public.

The main party to man the British Scientific Instrument Exhibition at Moscow, organized by the Scientific Instrument Manufacturers Association of Great Britain leave London Airport.



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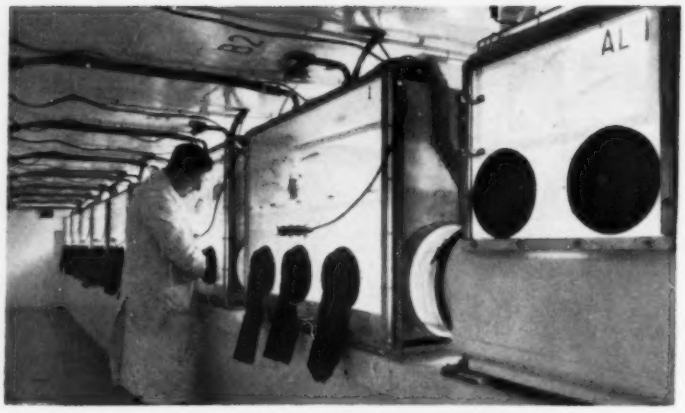
The Radiochemical Centre

A 24-page booklet has been issued by the U.K. Atomic Energy Authority (H.M.S.O., price 5s.) dealing with the Radiochemical Centre at Amersham—the organization within the Authority responsible for the production and marketing of radioisotopes.

This booklet describes the growth of the Centre from its beginnings in 1940 as a small laboratory for refining radium and producing self luminous paint to becoming a large scale scientific and commercial establishment providing facilities and services which are available to all countries throughout the world.

The organization of the Centre is briefly described and facts about the technology of isotope production are also given. The booklet also gives an outline of some of the many ways in which radioisotopes are used in medicine, industry and research.



The service corridor behind an alpha-beta-gamma 'hot' bench.

CATALOGUES, BROCHURES & LEAFLETS

Received from Manufacturers

Cambridge Publications.—Included in the recent leaflets issued by the Cambridge Instrument Co., Ltd. of 13 Grosvenor Place, London, S.W.1, are the following: *List* 97—describing the new general purpose polarograph which can be purchased in a simple and inexpensive form for manual plotting from a galvanometer and, later, by adding separate units, may be built up into an accurate and rapid pen-writing instrument. *List* 176—describing a range of thermometer regulators. *List* 309/1—describing an industrial gas analyser. *List* 304/1—describing the needle pyrometer which consists of a light portable indicator and a needle thermocouple which will penetrate below the surface of materials such as rubber and plastic to provide rapid and accurate temperature readings.

Duplex Filters.—A leaflet describing their complete range of Duplex filters has been published by Plenty & Son Ltd., of Eagle Iron Works, Newbury, Berks. These are complementary to the Simplex range (¼ in. up to 24 in.) which have been installed in refineries, gas works, coal mines, food factories, chemical plants, distilleries and ships throughout the world since their introduction four years ago.

Optical Aids.—This is the title of a new leaflet issued by Wray (Optical Works) Ltd., Bromley, Kent. It contains brief details of a binocular magnifier, inspection telescope, the Wrayflex on copying stand, microfilm reader, measuring magnifier and focusing magnifier.

Marconi Instrumentation.—The March 1960 issue of *Marconi Instrumentation* (Vol. 7, No. 5) published by Marconi Instruments Ltd., of St. Albans, Herts., contains articles on an electronic counter, a pH meter and apparatus designed for telegraph signal distortion analysis.

BTL Bulletin.—The May 1960 issue of the BTL Bulletin issued by Baird & Tatlock (London) Ltd., of Chadwell Heath, Essex, includes technical details of a wide variety of equipment manufactured by the company.

B.D.H. Leaflets.—New entries in the catalogue of British Drug Houses Ltd., of Poole, Dorset, are listed in the May, 1960 issue of their monthly leaflet. These are as follows: *Organic and Inorganic Chemicals.*—α-amylase and β-amylase; phloretin; *o*-phthalaldehyde.

Electronic Temperature Indicators.—A leaflet has been published describing the new electronic temperature indicators marketed by Electrothermal Engineering Ltd., of 270 Neville Road, London, E.7. The Tastotherm, measurement by contact for temperatures up to 400°C. (752°F.) is for medical research, clinical laboratory and industrial uses, and the Tastotherm Infra, measurement by radiation for temperatures up to 3,000°C. (5,432°F.) is for heavy industry, nuclear research and production processes.

New Springham Catalogues.—Latest publications issued by G. Springham and Co., Ltd., of Harlow New Town, Essex, are: 1. New catalogue detailing wider range of items, including automatic burettes, adaptors, Dean & Stark receivers, nitrometers, stopcock rails, etc. all fitted with interchangeable keys. 2. An addendum to the first catalogue detailing PTFE keys, fully interchangeable, with the above items. These keys are self-lubricating and chemically inert to almost all chemicals, solvents, etc. 3. Pamphlet detailing new and improved pattern of greaseless stopcock—essential where elimination of greases is required under conditions of high vacuum and wide temperature range.

Laboratory Chemicals.—The new edition of the B.D.H. Laboratory Chemicals catalogue, published by B.D.H. Laboratory Chemicals Division, Poole, Dorset, includes many substances of biochemical interest that have not appeared before. In all, it lists between 6,000 and 7,000 products and section 3 of the catalogue, formerly limited to amino acids, now also contains reference lists of other biochemical materials grouped for convenience in their various classes of carbohydrates, lipids, proteins, sterols, etc.

British Chemicals.—Laporte Industries Ltd. of 14 Hanover Square, London, W.1. have produced a magnificent full colour book—'The Raw Materials of Progress'—in which is illustrated the story of the group's activities, its history and development.

Laboratory PTFE Ware.—A new range of polytetrafluoroethylene beakers with a much lower pricing is being marketed by Jencons (Scientific) Ltd., of Mark Road, Hemel Hempstead, Herts. These are described in a new leaflet issued by the company which also contains details of PTFE tubing and rod which can now be bought in small supplies by laboratories. The company will be pleased to supply copies of these leaflets which give interesting technical data as well as prices.

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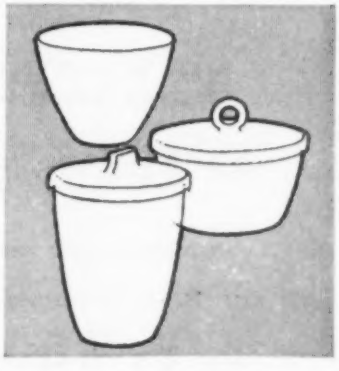

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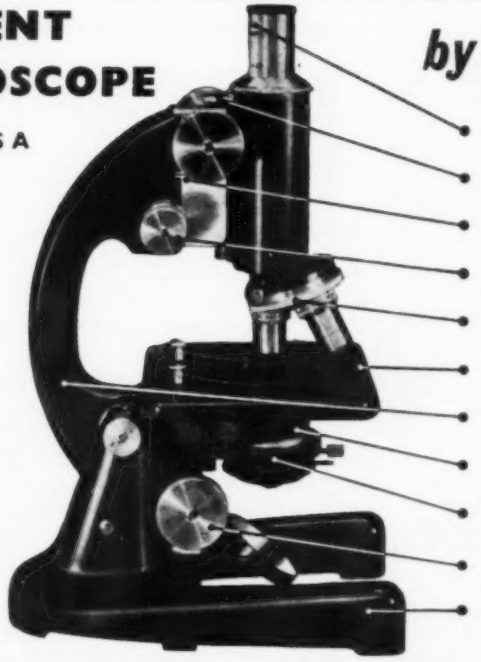

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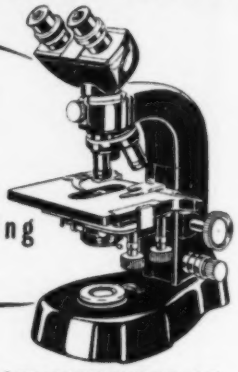

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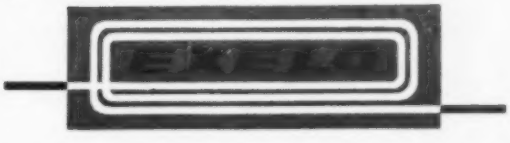

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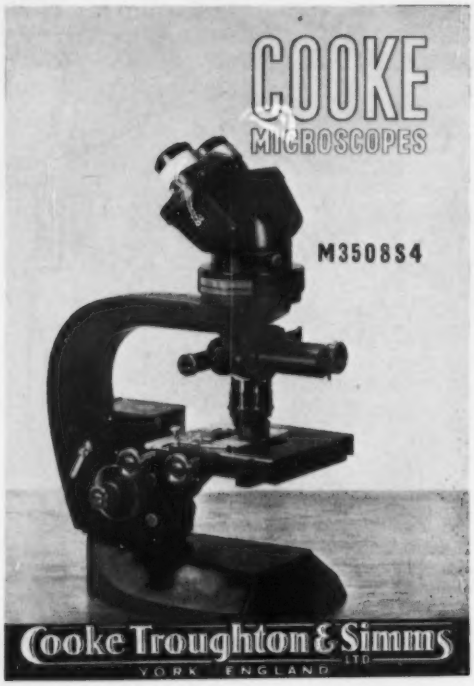

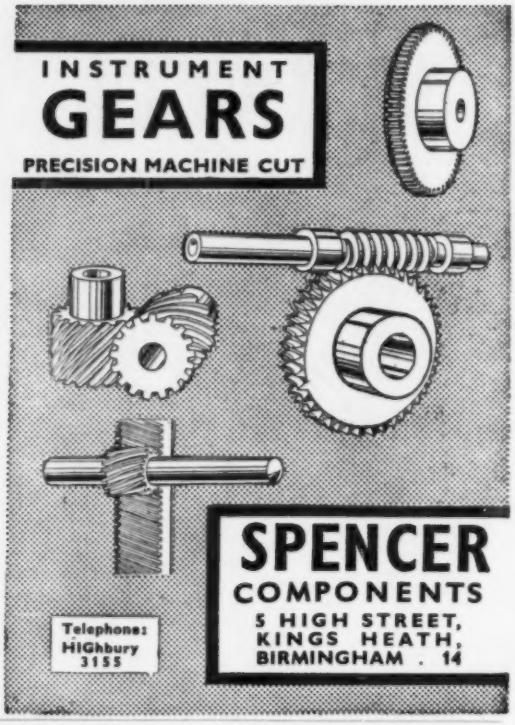

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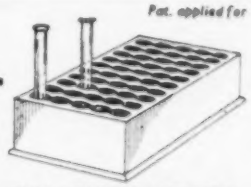

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